0632

INSTRUCTION MANUAL

# MODULINE® SERIES 2700

# GAS CHROMATOGRAPH

varian aerograph
2700 mitchell drive/walnut creek/california 94598/415-939-2400
specialists in chromatography and data handling systems

**MODEL 2740-10**
Publication No. 85-001118-66

The Model 2740-10 Instruction Manual includes the following modularized manuals:

| TITLE | PUBLICATION NO. |
|---|---|
| 2700 Basic Instrument | 85-001118-01 |
| Linear Temperature Programmer | 85-001118-02 |
| Dual/Differential Electrometer | 85-001118-06 |
| Alkali Flame Ionization Detector | 85-001118-11 |
| Flame Ionization Detector | 85-001118-12 |
| $Sc^{3}H$ Electron Capture Detector | 85-001118-17 |
| $^{63}Ni$ Electron Capture Detector | 85-001118-14 |
| Rotameter Gas Flow Controller | 85-001118-08 |
| Pressure Gauge Gas Flow Controller | 85-001118-09 |

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# ADDENDUM

**SUBJECT:** Toggle Flow Diverters
**INSTRUMENTS AFFECTED:** 1400, 2100, 2400, 2700, 2800 Series
**DATE:** October 1972

**ACTION:**
The optional flow diverter described in your instruction manual has been changed. The new style "toggle type" diverter (illustrated below) replaces the "push-pull" type previously used. Operation of the toggle flow diverter is as follows:

**NOTE: The following operational description should be substituted for the "push-pull" diverter operational description found in the manual.**

Divert Operation: Flip toggle handle toward outlet port. Monitor gas flow at outlet port.

Non-divert Operation: Flip toggle handle away from outlet port. Gas flow is normal, i.e., not diverted.

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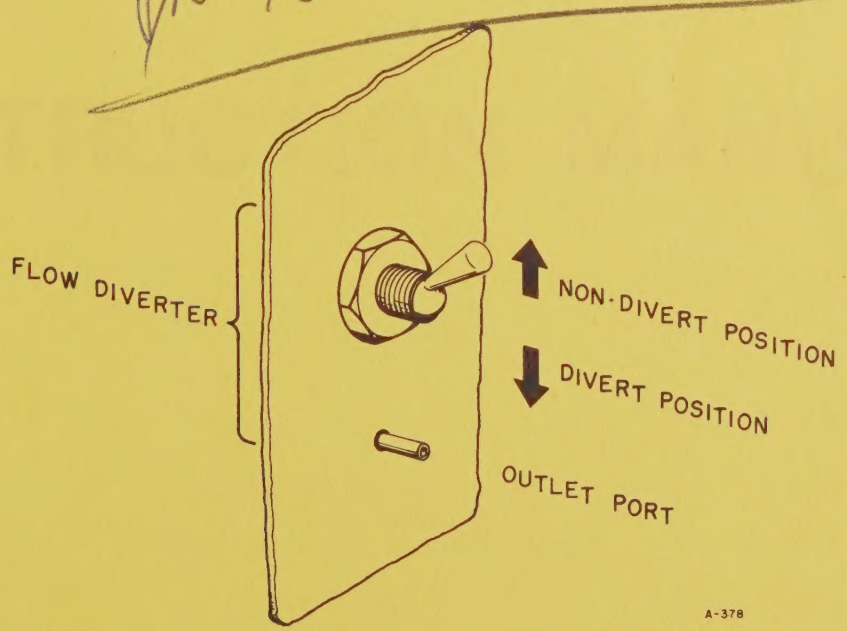

varian
instrument division
611 hansen way / palo alto / ca 94303 / u.s.a. / phone 415-493-8100

# AEROGRAPH®
# MODULINE® 2700
# SERIES BASIC
# INSTRUMENT

# INSTRUCTION MANUAL

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PUBLICATION NO. 85-001118-01

PRINTED IN U.S.A. AUGUST 1972

varian aerograph

# WARRANTY

Instruments and accessories sold by Varian Aerograph, unless otherwise specified, are warranted for a period of one (1) year against defects in materials and workmanship in accordance with the terms set forth below:

1. The warranty period begins on the date of shipment of the equipment from Varian Aerograph to the original purchaser.

2. This warranty applies only to internal electronic and mechanical parts and labor provided by Varian Aerograph under valid warranty claims. Certain parts such as batteries, fuses, glass accessories, septums, columns, chart paper, ink, etc., are expendable in normal use, and their service life is unpredictable. Such expendable items are not covered by this warranty.

3. Filaments of thermal conductivity detectors and thermistors for micro-absorption detectors are warranted for ninety (90) days.

4. All claims under this warranty must be received within the applicable warranty period by Varian Aerograph or its authorized representative and shall be subject to the standard Varian Aerograph Terms and Conditions of Sale.

5. All warranty service and replacement or repair of parts shall be limited to equipment malfunctions which, in the sole opinion of Varian Aerograph, are due or traceable to defects in original materials or workmanship. Instrument malfunctions caused by abuse or neglect of the equipment are expressly not covered by this warranty.

6. Varian Aerograph products are constructed of materials selected for maximum durability consistent with product performance under normal usage. However, certain chemicals, alone or in combination, may react with or otherwise damage the Varian Aerograph product or its components. Malfunction and/or damage resulting from chemical action are expressly not covered by this warranty.

7. In-warranty repaired or replacement instrument parts are warranted only for the remaining unexpired portion of the original warranty period applicable to the repaired or replaced parts.

8. Repair and replacement of parts and service performed after expiration of the applicable warranty period shall be charged to the purchaser at the then current prices for parts, labor and transportation.

9. Reasonable care must be used to avoid hazards. Varian Aerograph expressly disclaims responsibility for loss or damages caused by use of its products other than in accordance with proper operating procedures. Under no circumstances will Varian Aerograph be liable for incidental, consequential, or resulting loss or damage of any kind, howsoever caused. In no event will Varian Aerograph be liable for any sum greater than the purchase price received by Varian Aerograph for the product or products which are the subject of any claim or dispute.

10. Except as stated in Sections 1 through 9 above, Varian Aerograph makes no warranty, expressed or implied (either in fact or by operation of law), statutory or otherwise; and, except as stated in Sections 1 through 9 above, Varian Aerograph shall have no liability under any warranty, expressed or implied (either in fact or by operation of law), statutory or otherwise.

11. Statements made by any person, including representatives of Varian Aerograph, which are inconsistent or in conflict with the terms of this warranty shall not be binding upon Varian Aerograph unless reduced to writing and approved by an officer of Varian Aerograph.

12. This warranty shall be governed by the law of California.

## SAFETY PRECAUTIONS AND OPERATING INSTRUCTIONS

This instruction manual is provided to help you establish operating conditions which will permit efficient use of your equipment. You will notice that the manual describes special considerations and precautions in the form of notes, cautions, and warnings, that not only aid you in obtaining the best performance from your equipment but in calling to your attention potential hazards (e.g., electrical, mechanical, etc.) that might cause personal injury, and how to avoid these hazards. It is important, therefore, that you operate your equipment in accordance with this instruction manual and additional information which may be provided by Varian Instrument Division, Aerograph® Products. Address any questions regarding the safe and proper use of your instrument to:

Varian Instrument Division, Aerograph® Products
611 Hansen Way
Palo Alto, California 94303

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## SPARE PARTS AVAILABILITY

It is the policy of the Varian Instrument Division to provide operational spare parts for any instrument and major accessory for a period of five (5) years after shipment of the final production run of that instrument. Spare parts will be available after this five (5) year period but on an "as available" basis.

Operational spare parts are defined as those individual electrical or mechanical parts that are susceptible to failure during their normal operation. Examples include resistors, inductors, capacitors, semi-conductor devices of all kinds, relays, lamps, temperature probes, detector elements, motors, etc.

Sheet metal parts, structural members or assemblies, and castings, printed circuit cards, and functional modules are normally capable of being rebuilt to like-new condition throughout their useful life and therefore will be supplied on an "as available" basis only after the final production run of the instrument.

The repair and/or reconditioning of instruments and accessories is handled on a time and materials basis after expiration of the Warranty period. Please contact your nearest Varian Instrument Division Sales and Service Office for a quotation.

6/20/1972

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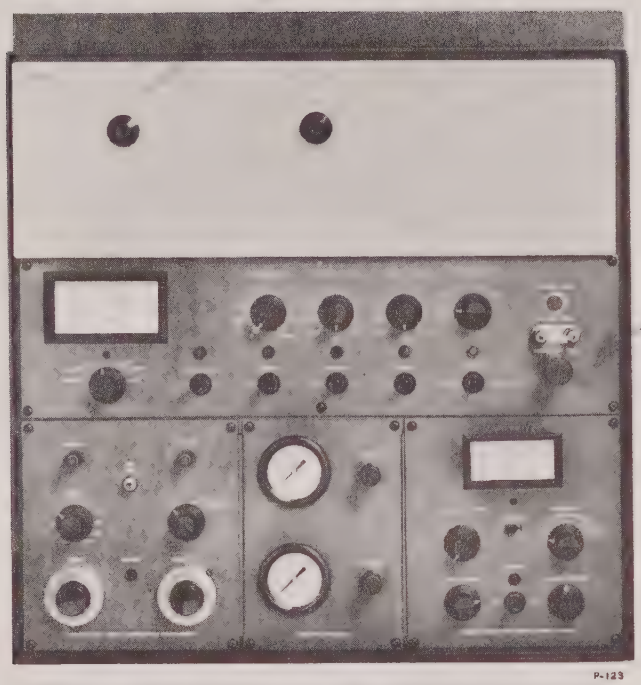
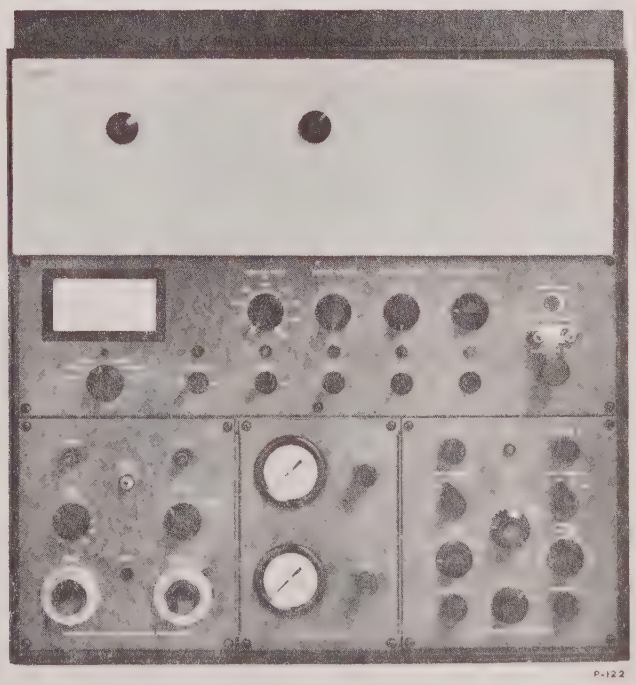



2720 SERIES




2740 SERIES

# SECTION 1
# INTRODUCTION

## 1.1 GENERAL

The Moduline® 2700 Series instruments are dual column gas chromatographs. The instruments are available with a selection of detectors, temperature programmers, gas flow controllers, etc. Table 1-1 lists the various models available in the 2700 Series and the components or modules included as part of the instrument. A description of the various components (modules) comprising your instrument will be covered in individual manuals included with this publication.

All thermal regions, i.e., injectors, column oven, and detector oven, in the instrument are thermally isolated and have individual temperature controls. Differential flow controllers are also included in either flow control module to provide a constant flow of carrier gas. For instruments equipped with flame ionization detectors, fine metering valves are included to permit individual adjustment of air and hydrogen supply to each detector.

## 1.2 SPECIFICATIONS

**Column Oven**

Size: Height—8" (20 cm); Width—13" (33 cm); Depth—10.5" (27 cm); Volume—1090 $in^3$ (17,820 $cm^3$).
Temperature Range: Ambient to 400°C (-100°C to 400°C option).
Cooling Rate: 400 to 100°C in 3 minutes.
Temperature Limit Protection: Adjustable (150 to 400°C) temperature limiting device prevents overheating of columns.
Air Circulation: Internal centrifugal blower.
Construction: Stainless steel with heavy-duty insulation.

**Detector Oven**

Temperature Range: Ambient to 400°C.
Stability: ± 0.5°C by full proportional control.
Construction: Cast aluminum.
Temperature Limit Switch: Limits temperature when $^3H$ detectors are used.

**Injectors**

Temperature Range: Ambient to 400°C.
Injection Methods: Flash vaporization or on-column; provisions for glass injector liners to provide nonreactive surface.
Construction: Two machined stainless steel tubes.
Temperature Control: Independent temperature control and pyrometer readout for each injector.

**Flow System**

Carrier Gas: Single input to on-off valve.
Hydrogen and Air: Individual $H_2$ and air connections with separate needle valves for controlling $H_2$ and Air to each ionization detector (flow diverters optional).

**Temperature Monitoring**

Indicator: Thermocouple pyrometer with parallax correction mirror.
Range: 0 to 400°C with accuracy of ± 2% full scale.
Temperature Readout: Injectors, column oven, and detector oven. Provisions for monitoring two external thermocouples (10-ohm iron-constantan).

**Output**

Recorder: Aerograph, Model 10, 25, 30 or equivalent, 1 mV, 1 sec, full scale response with floating input.
Integrator: Aerograph Model 477, 480, 481 or equivalent, 0.1 or 1 volt full scale range with floating input.
Data System: Aerograph CDS 200 Series or equivalent, 0-10 volt input range.

**Power Requirements**

Domestic: 103 to 127 Vac, 60 Hz, 20 amps (max).
Other: 90 to 145 Vac transformer selectable, 50/60 Hz, 20 amps (max). 180 to 265 Vac transformer selectable, 50/60 Hz, 10 amps (max).

**Dimensions**

Width: 19 1/2 inches (49.5 cm).
Height: 21 7/8 inches (55.6 cm).
Depth: 21 1/2 inches (54.6 cm).

**Weight**

Net: 130 lbs (59 kg).
Gross: 155 lbs (70 kg).

TABLE 1-1 2700 SERIES DUAL COLUMN GAS CHROMATOGRAPHS

| MODELS | DETECTORS | | ELECTRONIC MODULES | | | | | FLOW CONTROLLERS | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Dual[1] Flame Ionization | Dual Thermal Conductivity | Linear Temperature Programmer (LP) | Isothermal Proportional Controller (IPC) | Manual Controller | Dual/ Differential Electrometer | Thermal Conductivity Bridge | Pressure Gauge Module | Rotameter Module | Needle Valves $H^2$ & Air | Flow Diverters |
| 2720-10 | | X | X | | | | X | X | O | | |
| 2720-20 | | X | | X | | | X | X | O | | |
| 2720-50 | | X | | | X | | X | X | O | | |
| 2740-10 | X | | X | | | X | | X | O | X | O |
| 2740-20 | X | | | X | | X | | X | O | X | O |
| 2740-50 | X | | | | X | X | | X | O | X | O |

1. Standard detectors are Flame Ionization. Electron Capture, ($^3H$ and $^{63}Ni$) and AFID detectors are optional and directly interchangeable.

X — Standard Items

O — Optional Items

# SECTION 2
# INSTALLATION

## 2.1 UNPACKING

When unpacking your instrument, check carefully to ensure that all components ordered have been supplied. Refer to the accessory packing list enclosed with your instrument. If there are discrepancies, notify Varian Aerograph.

NOTE

**If any damage is found, notify the Carrier and Varian Instrument Division, Aerograph® Products immediately. Do not return the instrument to the factory unless authorized by a Varian representative. Be sure to include written authorization from representative or factory with instrument when returning to factory.**

The packing box should be retained for use should the instrument need to be returned for repair or modification.

## 2.2 LOCATION

Your instrument should be installed in a location where it will not be subject to abrupt temperature changes, excessive vibration, or strong air currents (such as under a heater or air conditioning blower). If gas supplies are not already available, make sure that there is adequate space for their installation. Place the instrument in a location which will provide a minimum of four inches of clearance behind the instrument. A minimum of twelve inches above the instrument is necessary to permit opening and closing the oven door.

## 2.3 POWER REQUIREMENTS

All 2700 Series instruments require a single-phase (50 or 60 Hz) power line capable of providing up to 20 amps at 90 to 145 Vac or 10 amps at 180 to 265 Vac. Instruments are ordered according to the power line frequency and voltage available at the installation site.

All instruments sold outside Continental North America contain a multi-voltage tap transformer. This transformer permits the instrument to be wired for operation over a voltage range of 90 to 265 Vac. However, instruments are shipped wired (appropriate voltage tap selected on transformer) for the nominal voltage available at the installation site. The voltage tap selected can be observed on a dial plate visible through the cutout located on the rear power panel just above the power cord. The voltage tap selected is the one that comes closest to that available at the installation site. All instruments sold inside Continental North America do not have the multi-voltage tap transformer unless specified in the original order. These instruments are normally setup for 115 Vac @ 60 Hz.

If your instrument, i.e., one equipped with the multi-voltage tap transformer, is to be operated on a different line voltage from that originally selected, the appropriate tap on the transformer must be selected to accept the new line voltage.

NOTE

**If you desire to operate the instrument over a voltage range of 180 to 265 Vac but the instrument is wired for operation over the 90 to 145 Vac range, or vice versa, additional wiring changes beyond changing the transformer tap are required. Please contact your Varian Representative for additional information on wiring changes.**

The transformer has a series of numbered taps which correspond to a given voltage as follows: Tap #0, Neutral; #1, 100 V; #2, 110 V; #3, 115 V; #4, 120 V; #5, 130 V; #6, 200 V; #7, 220 V; #8, 240 V. Refer to Figure 2-1 for location of taps on transformer. The rear power panel must be lowered enough to gain access to transformer. The tap

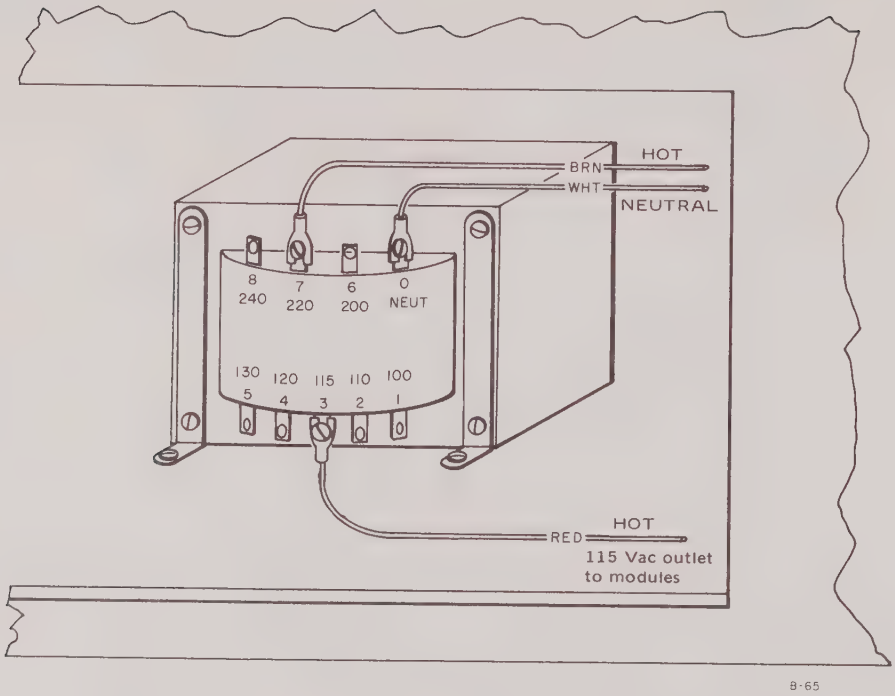



FIGURE 2-1 LOCATION OF TAPS ON POWER TRANSFORMER (REAR POWER PANEL REMOVED)

which comes closest to the nominal line voltage should be used. All instruments will operate within ±10% of the voltage tap selected on the transformer. Move the brown wire to the appropriate tap as previously designated.

**WARNING**

**To avoid a possible shock hazard, always disconnect power cord from ac receptacle when removing rear power panel.**

The ac power cord on all 2700 Series instruments is terminated with a male 3-prong straight blade plug. A matching 3-prong straight blade receptacle is required for the ac outlet. Make sure the outlet receptacle used to connect your instrument to ac power has the correct polarity. This outlet receptacle should be wired as shown in Figure 2-2. The proper polarity can easily be checked with a VOM or by observing the MAIN POWER switch indicator. Should the indicator stay lit with the switch in the off position, the hot and neutral lines are reversed. Line voltage will be measured between hot and ground connections and zero volts between neutral and ground connections. Also, make sure the instrument is connected to a good earth ground either through the power receptacle or with a separate wire connected to the CHASSIS binding post on the rear power panel of the instrument. The instrument will not function properly unless properly grounded.

**WARNING**

**If the outlet receptacle used with the instrument has the ac polarity reversed, the instrument will not function properly and a shock hazard may result.**

To make sure all fuses are good and that power is applied to all modules, plug the ac line cord into the appropriate receptacle, then proceed as follows:

NOTE

**When performing the following steps, disregard discussion of modules which are not installed in your instrument.**

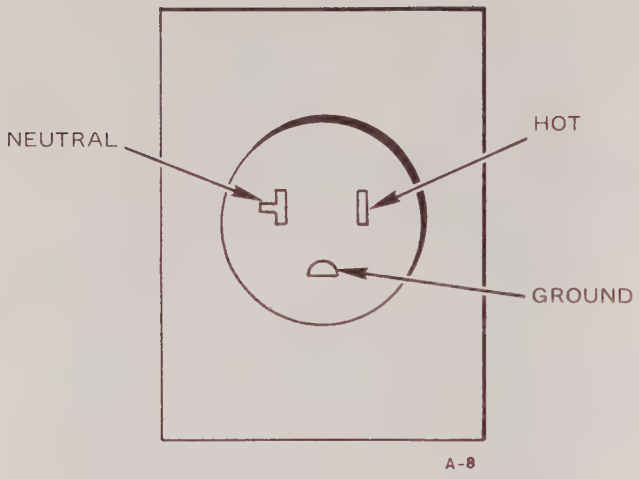



FIGURE 2-2 OUTPUT POLARITY OF STRAIGHT—BLADE RECEPTACLE

1) Turn FILAMENT CURRENT control on TC Bridge Module to full counterclockwise position. Do not allow current to flow in TC filaments without the presence of carrier gas, to prevent possible damage to filaments.

2) Turn on MAIN POWER switch (located on main control panel) to supply power to all circuits on main control panel and Electrometer Module. Light above switch should illuminate indicating power is on. POWER lamp on Electrometer module should also light indicating power is on.

3) Turn on POWER switch on temperature programmer module. This module has an independent switch for turning power on or off. Light above switch should illuminate indicating power is on.

4) Turn on POWER switch on TC Bridge Module. Light above switch should illuminate indicating power is on. Since no carrier gas is flowing through TC cell, switch must be turned off immediately to prevent any possible damage to TC filaments.

5) Turn on remaining switches to make sure associated circuits receive power.

6) Turn all switches off before proceeding with the installation of your instrument.

## 2.4 COLUMN INSTALLATION

Unless specified, the 2740 Series instruments are supplied with 1/8-inch by 5-foot SE-30 columns set up for on-column injection. The 2720 Series instruments are supplied with 1/4-inch by 5-foot SE-30 columns set up for flash vaporized injection. The appropriate injector insert (refer to Figure 2-3) should be used for connecting either 1/4- or 1/8-inch columns. These inserts eliminate dead volume in the injector and may easily be removed for cleaning.

When on-column injections with 1/4-inch columns are desired, the insert should be removed and the end of the column inserted into the injector to a point about 1/4-inch behind the septum before swaging on the ferrules. On-column injections with 1/8-inch columns require the installation of the 1/4-inch insert with the 1/8-inch ID. The 1/8-inch column is inserted into the injector insert to a point about 1/4-inch behind the septum before swaging on the ferrules.

Optional or replacement columns ordered from Varian will not have the fittings attached. The fittings for each column are in a plastic package. This method of shipment permits column installation for either flash vaporization or on-column injection.

Before installing any columns, make sure the appropriate injector insert is installed if you are using 1/8-inch columns or 1/4-inch columns set up for flash vaporization. No insert is used when setting up a 1/4-inch column for on-column operation. Refer to Figure 2-3 for set up of injectors.

When you plan to use the TC detector in the 2720 Series instruments, you must install two columns; i.e., two packed columns or one packed column and one restrictor (blank) column. The packed column is usually installed in flow system B (analytical) and the restrictor column in flow system A (reference). Reverse order is acceptable. When installing two packed columns or matched columns, it makes no difference in which flow system either column is installed.

Depending on whether you are installing one or two columns, the installation procedure is the same.

Install column(s) as follows:

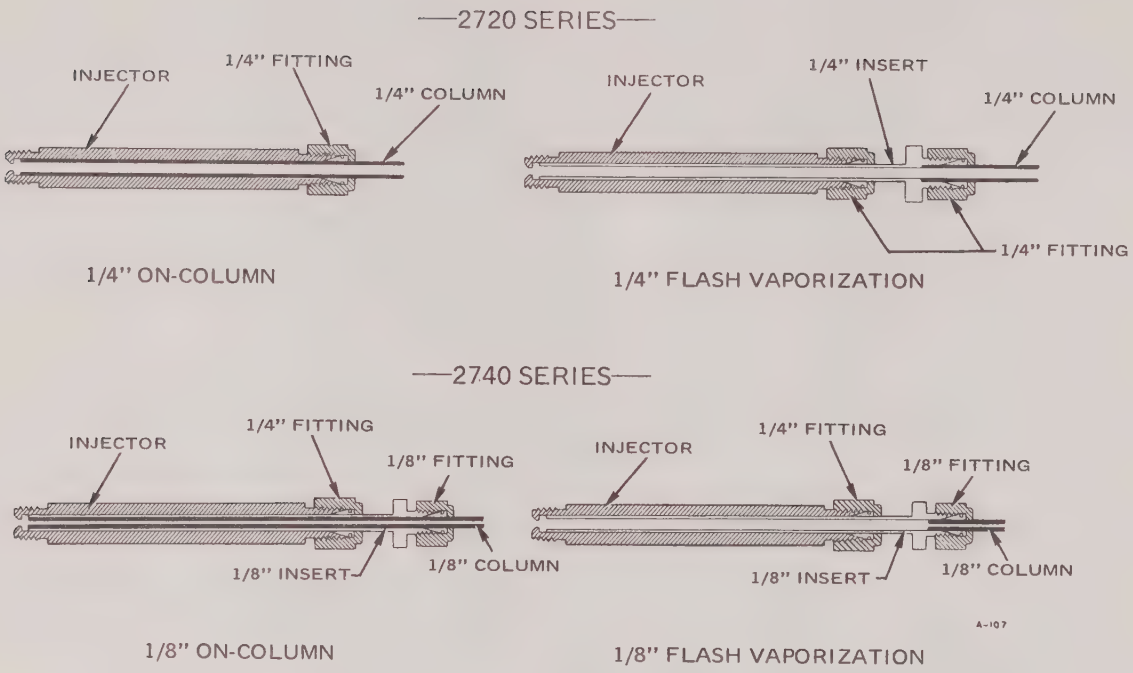



**FIGURE 2-3 COLUMN INSTALLED IN INJECTOR FOR FLASH VAPORIZED OR ON—COLUMN INJECTION**

1) Open column-oven door.

2) Make sure injector is set up as discussed above.

3) Place column in oven and orient as shown in Figures 2-4 and 2-5. Figure 2-4 shows a Model 2720 and Figure 2-5 shows a Model 2740.

4) Secure column to injector insert (or injector). Two wrenches must be used when connecting a column to an injector insert to prevent damage to injector insert fitting. Use one wrench to prevent injector insert from turning while tightening the column fitting with the other wrench. Repeat this procedure if second column is to be installed. Tighten fitting approximately 1-1/4 turns past finger tightness.

5) Secure other end of column to crossover fitting. Tighten fitting approximately 1-1/4 turns past finger tightness. Repeat this procedure if second column is to be installed.

6) Close column-oven door.

## 2.5 GAS SOURCES AND CONNECTIONS

The following gas sources and connections are necessary for operation of your instrument: A carrier gas cylinder of either He or $N_2$ (FID), He or $H_2$ (TC), or $N_2$ ($^3H$, $^{63}Ni$ and AFID). A 100 psi pressure regulator is required on the cylinder. In addition to carrier gas, air and hydrogen are required for operation of the FID and AFID detectors. When operating an AFID detector, the regulation of air and hydrogen is very important. We recommend the use of our Model 403 Dual Flow Controller.

Hydrogen may be supplied from a cylinder or a Model 9652 Hydrogen Generator. An air cylinder must be used with cylinder hydrogen or the Model 9652 Hydrogen Generator.

Nitrogen carrier gas of high purity must be used when operating $^3H$ and $^{63}Ni$ detectors. This high purity nitrogen must be especially free of oxygen and water vapor (less than 5 ppm), since these materials are strong electron adsorbers. Filters, i.e., molecular sieve, particulate, oxygen, are available

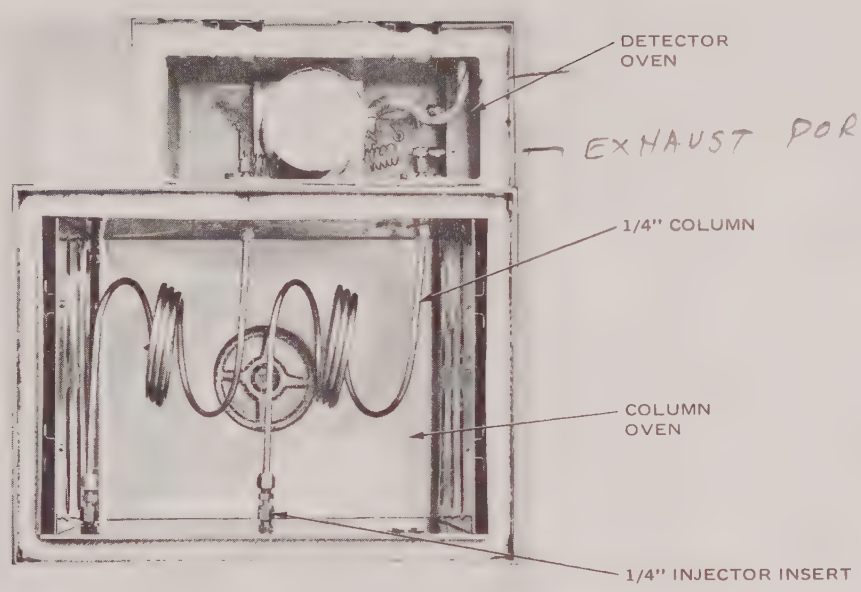



FIGURE 2-4 2720 SERIES COLUMN INSTALLATION

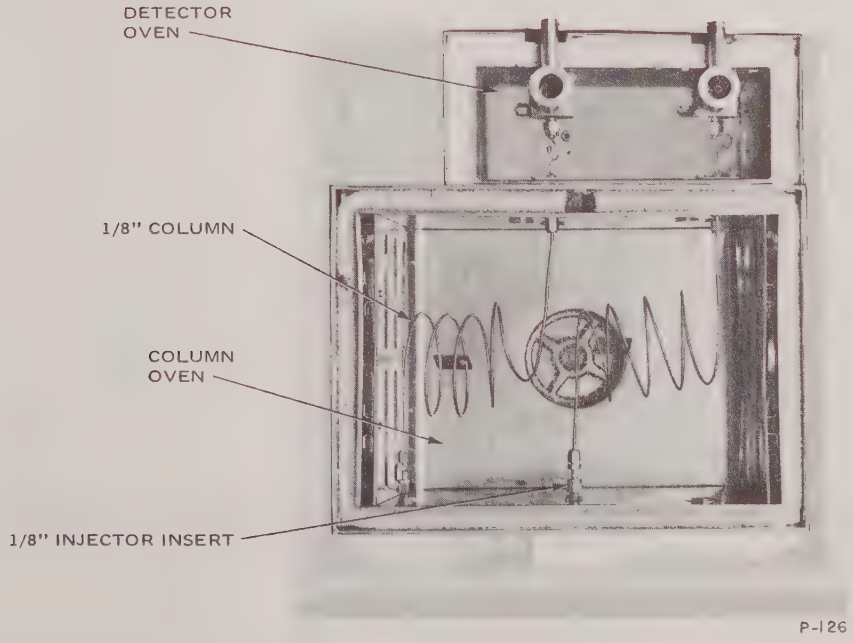



FIGURE 2-5 2740 SERIES COLUMN INSTALLATION

from Varian Aerograph for inserting between carrier gas tank and instrument. The use of the filters is recommended, especially when using the $^{3}H$ and $^{63}Ni$ detectors.

Refer to the following paragraphs for connecting the gas supplies to your instrument.

### 2.5.1 Carrier Gas Connections (2720 and 2740 Series)

Supplied as standard with your instrument is a 1/8-inch by 5-foot length of tubing. This line is supplied with a 1/4-inch pipe to 1/8-inch pipe fitting for connecting line to pressure regulator and a 1/8-inch fitting with nut and ferrule for connecting other end of line to CARRIER GAS input fitting on side of instrument. Install line by connecting the 1/4-inch fitting to the pressure regulator on the carrier gas cylinder and the 1/8-inch fitting to the CARRIER GAS input fitting on side of instrument.

### 2.5.2 Hydrogen Connections (2740 Series)

Supplied as standard accessory with your instrument is a 1/8-inch by 5-foot length of tubing. This line is supplied with a 1/4-inch pipe to 1/8-inch pipe fitting for connecting line to pressure regulator and a 1/8-inch fitting with nut and ferrule for connecting other end of line to HYDROGEN fitting on side of instrument. Install line by connecting the 1/4-inch fitting to the pressure regulator on the hydrogen cylinder and the 1/8-inch fitting with nut and ferrule to the HYDROGEN input fitting on side of instrument.

If you are using a Model 9652 Elhygen Hydrogen Generator, the appropriate tubing and fittings are supplied with the generator for connecting the generator to your instrument.

### 2.5.3 Air Connections (2740 Series)

Air must be supplied from a cylinder. Supplied with your instrument is a 1/8 by 5-foot length of tubing. This line is supplied with a 1/4-inch pipe to 1/8-inch pipe fitting for connecting line to pressure regulator and a 1/8-inch fitting with nut and ferrule for connecting other end of line to AIR fitting on side of instrument. Install line by connecting the 1/4-inch fitting to the pressure regulator on the air cylinder and the 1/8-inch fitting with nut and ferrule to the AIR input fitting on side of instrument.

## 2.6 GAS LEAK CHECKS

To ensure proper operation of your instrument, carrier gas and hydrogen (where applicable) flow systems and input lines should be leak checked. A leak check of input gas lines should be made each time a new tank supply is connected to the instrument.

All 2740 Series instruments are shipped with a special detector base plug installed in each ionization detector base. This plug must be installed to make hydrogen (FID and AFID detectors only) and carrier gas leak checks. The small tube extending from the plug also provides a convenient place for monitoring air flow rates. After making leak checks, air flow rates (FID and AFID detectors only) should be established; therefore, leave plug installed and refer to paragraph 3.4 on air flow measurements. After air flow measurements are completed, the plug can be removed and you can proceed with detector installation.

**NOTE**

**If your instrument is equipped with flow diverters, the detector base plug is not required for air flow measurement.**

### 2.6.1 Carrier Gas Input Line Leak Check

A leak check of the carrier gas input line is necessary for all 2700 Series instruments. Check for leaks as follows:

1) Turn CARRIER GAS FLOW control on main control panel fully clockwise (off).

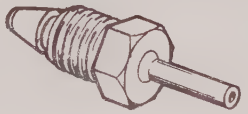
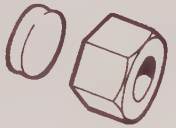
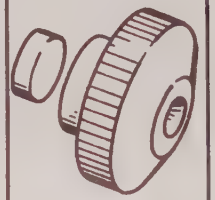
2) Adjust regulator control on carrier gas tank for about 65 psi. Allow pressure to stabilize. Turn off regulator and observe gauge. Pressure should not drop more than a few psi, indicating no leaks between cylinder and instrument. If there is a constant drop in pressure, check fitting at regulator and instrument for leaks. Correct any leak(s).

### 2.6.2 Carrier Gas Flow System Leak Check for TC Equipped Instruments (Series 2720)

The carrier gas flow system of all 2720 Series instruments must be leak checked. Proceed as follows:

1) Prepare instrument for leak check as outlined in Table 2-1.

TABLE 2-1
LEAK CHECK COMPONENTS

| Series being leak checked | Cap off ionization detector bases with detector base plug. Plugs are installed when instrument is shipped from factory.  A-136 | | Cap off collector tubes. Use collector nuts with one injector septum installed in each nut.  A-136 | | Cap off hydrogen input fitting. Use 1/8" blank-off nut. A-136 | | Install injector nut and septum on each injector port.  A-136 |
|---|---|---|---|---|---|---|---|
| | DETECTION SYSTEM | | DETECTION SYSTEM | | DETECTION SYSTEM | | |
| | TC | IONIZATION | TC | IONIZATION | TC | IONIZATION | ——— |
| 2720 | N.A. | N.A. | YES | N.A. | N.A. | N.A. | YES |
| 2740 | N.A. | YES | N.A. | N.A. | N.A. | YES | YES |

N.A.: Not Applicable

2) Make sure columns are properly installed.

3) Set INJECTOR A and B control valves to full counterclockwise (on) position. Adjust CARRIER GAS FLOW control to full counterclockwise (on) position.

4) Adjust regulator on carrier gas tank to 65 psi. Allow pressure to stabilize.

5) Turn off tank valve on carrier gas tank and observe low pressure gauge. Pressure should not drop more than a few psi, indicating no leaks. If there is a constant drop in pressure, check plumbing connections, e.g., column connections, around injector nuts and septa, etc., to locate leak(s). Correct any leak(s).

6) Restore instrument to preleak check condition.

**2.6.3 Carrier Gas and Hydrogen Flow System Leak Check for Ionization Detector Equipped Instruments (Series 2740)**

A leak check of the carrier gas and hydrogen flow system is necessary for all 2740 Series instruments. Since the carrier gas and hydrogen mix in the detector base, their respective flow systems can be checked simultaneously. However, the carrier gas and hydrogen flow systems can be isolated when operating, e.g., $^{3}H$/FI or $^{63}Ni$/FI combinations, by turning off the hydrogen supply to the appropriate base via the DETECTOR HYDROGEN A or B control valve. Proceed as follows:

— CAUTION —

**To prevent damage to valves, do not overtighten in the off position.**

1) Prepare instrument for leak check as outlined in Table 2-1.

2) Make sure columns are properly installed.

3) Set INJECTOR A and B control valves to full counterclockwise (on) position.

4) Set DETECTOR HYDROGEN A and B control valves to full counterclockwise (on) position.

NOTE

**For instruments equipped with flow diverters, set $H_2$ FLOW DIVERTERS to off (pulled out) position.**

5) Adjust CARRIER GAS FLOW control on main control panel to full counterclockwise (on) position.

6) Adjust pressure regulator on carrier gas tank for 65 psi. Allow pressure to stabilize.

7) Turn off tank valve on carrier gas tank and observe low pressure gauge. Pressure should not drop more than a few psi, indicating no leaks. If there is a constant drop in pressure, check plumbing connections, e.g., column connections, around injector nuts and septa, at the connections to the various control valves, etc., to locate leak(s). Correct any leak(s).

8) Restore instrument to preleak check condition.

NOTE

**If you are going to make a hydrogen tank supply line leak check, leave instrument setup as above, then refer to paragraph 2.6.4.**

**2.6.4 Hydrogen Tank Supply Line Leak Check (Series 2740)**

This procedure applies only to instruments using tank hydrogen. To check hydrogen tank supply line for leaks, proceed as follows:

1) Make sure instrument is setup as per paragraph 2.6.3, then remove cap-off fitting from HYDROGEN fitting and connect hydrogen supply line to fitting.

2) Turn off regulator at carrier gas tank.

3) Set regulator on hydrogen tank to 10 psi. Allow pressure to stabilize.

4) Turn off tank valve on hydrogen tank and observe low pressure gauge. Pressure should not drop more than a few psi, indicating no leaks between tank supply and instrument. If

a constant drop in pressure occurs, check plumbing connections at pressure regulator and HYDROGEN fitting on rear panel of instrument. Correct any leak(s). There should be no leaks inside instrument since hydrogen and carrier gas systems were checked for leaks in paragraph 2.6.3.

5) Relieve pressure by removing detector base plugs.

FIGURE 2-6 LOCATION OF RECORDER/INTEGRATOR AND COMPUTER TERMINAL STRIPS

## 2.7 DETECTOR INSTALLATION AND REMOVAL PROCEDURES

Installation and removal procedures for the detectors included with your instrument can be found in the detector manual(s) included with this publication.

## 2.8 RECORDER/INTEGRATOR CABLE CONNECTIONS

Interconnection of the recorder or integrator with the instrument is via a shielded cable supplied with your instrument. There are two cables supplied with the 2740 Series instruments and one cable supplied with the 2720 Series instruments. Each shielded cable terminates (both ends) in 3 leads, i.e., clear, black, and green (shield). One end of each cable has clips with bent ends and the other end has clips that are not bent. The clips with the bent ends must be connected to the RECORDER/INTEGRATOR terminal strip at the rear of the instrument. The remaining end of the cable connects to a recorder or integrator.

Locate RECORDER/INTEGRATOR terminal strip at rear of instrument from Figure 2-6, then make connections as outlined below.

**• ELECT A/ELECT B Connections (2740)**
Connect end of cable with bent clips to RECORDER/INTEGRATOR terminals marked ELECT A. Connect clear lead to +, black lead to -, and green (shield) lead to ground. Connect other end of cable to recorder, or integrator, observing the same color coding-to-polarity arrangement outlined above.

A signal will be present an ELECT A output terminals for all modes of Electrometer operation, e.g., A, B, A-B, B-A, FI-FI, and FI-EC. For the FI-FI and FI-EC modes, a signal will be present from channel A only at the ELECT A terminals. The signal from channel B will be present only at the ELECT B terminals. This means that a second cable must be connected to ELECT B terminals and the second channel of a dual channel recorder or second integrator. Connection of the cable to ELECT B terminals is the same as for ELECT A with the same color coding-to-polarity arrangement.

**• TC Bridge Connections (2720)**
If you are using a TC detector, only one cable is necessary for connecting the instrument to the recorder or integrator. Connect end of cable with bent clips to RECORDER/INTEGRATOR terminals marked TC. Connect clear lead to +, black lead to -, and green (shield) lead to ground. Connect other end of cable to recorder or integrator observing the same color coding-to-polarity arrangement as outlined above.

## 2.9 COMPUTER CABLE CONNECTIONS

Interconnection of a computer to the instrument is possible via the COMPUTER terminal strip shown in Figure 2-6. Signal distribution at the terminals is the same as that of the RECORDER/INTEGRATOR terminal strip. Refer to Table 3-2

for additional information on output signals available at the terminal strip.

## 2.10 COLUMN CONDITIONING

Standard columns supplied with the instrument have been preconditioned. However, all new columns should be preconditioned before being used. Refer to paragraph 3.5.1 for column conditioning techniques.

# SECTION 3
# OPERATION

## 3.1 GENERAL

This section of the manual discusses set up and operation of the basic 2700 Series instrument. The basic 2700 Series instrument includes control panels which control the overall functioning of the instrument. Included with the basic instrument are a number of modules which control specific functions of the instrument. The set up and operation of each module is described in separate manuals. These manuals are included with this manual — all manuals comprising a package reflecting your particular instrument.

## 3.2 MAIN CONTROL PANEL

The main control panel provides a central location for controlling ac power to the entire instrument, temperature control of injectors and detector, and temperature monitoring of all thermal regions. Refer to Figure 3-1 and Table 3-1 for location and description, respectively, of each control found on the main control panel.

FIGURE 3-1 MAIN CONTROL PANEL

TABLE 3-1
MAIN PANEL OPERATING CONTROLS

| | |
|---|---|
| TEMPERATURE READ-OUT Selector | Selects region to be monitored by pyrometer. The accessory and auxiliary positions permit addition of thermocouples to monitor accessory temperatures. |
| MAIN POWER Switch | Turns power on or off to entire instrument. Light above switch turns on when power is on. |
| INJECTOR A and B Switches | Turns power on or off to appropriate injector heater and controller. Lights above each switch turn on when power is on. |
| DETECTOR Switch | Turns power on or off to detector heater. Light above switch turns on when power is on. |

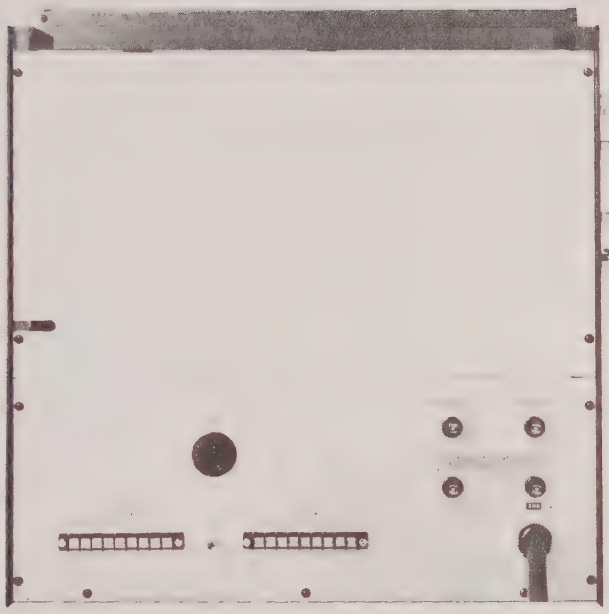
TABLE 3-1 MAIN PANEL OPERATING CONTROLS (cont'd)

| | |
|---|---|
| COLUMN OVEN Switch | OFF Position: Turns off column-oven fan and prevents heat and/or coolant from being applied to column oven.<br><br>FAN Only Position: Turns on column-oven fan only.<br><br>OVEN & FAN Position: Turns on column-oven fan and permits heat and/or coolant to be applied to column oven. |
| COLUMN SAFETY LIMIT °C Control | Prevents overheating of column oven. Automatically turns off column-oven heater when temperature reaches set limit. COLUMN OVEN light turns off when COLUMN SAFETY LIMIT °C setting is reached. Control cannot be accurately set below 150°C. |
| DETECTOR TEMP °C Control | Controls temperature of detector oven. Operation of heater control is indicated by light above DETECTOR switch. Temperature settings are indicated on panel around control. |
| INJECTOR A and B TEMP. Controls | Controls temperature of associated injector tube. Operation of heater control is indicated by appropriate INJECTOR light turning on. See Figure 3-6 for appropriate temperature setting. |
| Pyrometer | Indicates temperature of region selected by TEMPERATURE READOUT selector. |
| SAMPLE VALVE Access Hole | This access hole (located on Main Control Panel) will permit installation of optional 6-way or 7-way Gas Sampling Valve. Other mounting holes are located on right side of instrument for installing manifold section of valve. Valve is usually installed in B-flow system. |
| ACCESSORY GAS FLOW Fitting | This fitting (located on Main Control Panel) permits access to flow-controlled carrier gas between B-injector control valve and injector. A 1/8-inch stainless steel bypass tube connects these fittings to pass the carrier gas when no accessory is used. |
| CARRIER GAS FLOW Control Valve | Turns carrier gas supply to both flow controllers on or off. Use when changing columns, septums, etc. Turn fully counterclockwise to open, fully clockwise to close. |

## 3.3 POWER PANEL

The power panel provides a central location for all fuses, ac power input, recorder/integrator outputs, computer outputs, and gas inputs to your instrument. Depending on the particular instrument you have, certain cables, fuses, gas input connections may or may not be included. Figure 3-2 illustrates the power panels of the 2700 Series instrument. Refer to Table 3-2 for description of components found on power panels.

2720 SERIES



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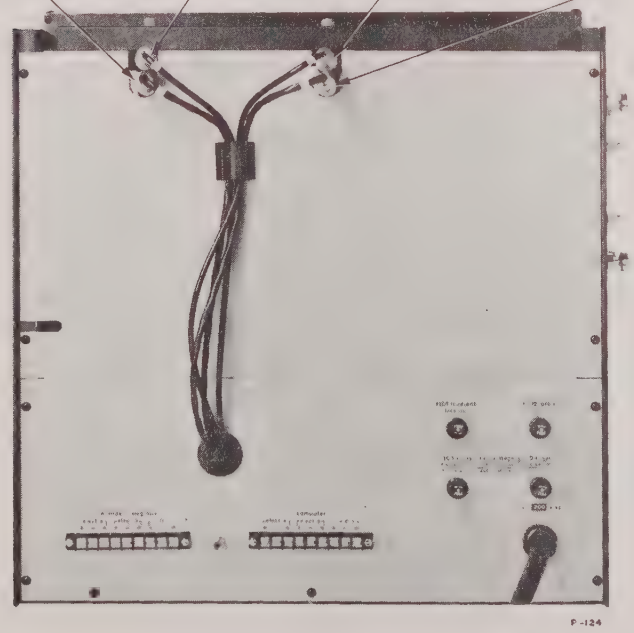
TYPICAL FID & AFID
CABLE CONNECTIONS*

CHANNEL B CELL VOLTAGE CABLE (2-COND CABLE)

CHANNEL B SIGNAL CABLE (1-COND CABLE)

CHANNEL A SIGNAL CABLE (1-COND CABLE)

CHANNEL B CELL VOLTAGE CABLE (2-COND CABLE)



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2740 SERIES

*NOTE: The position of the signal and cell voltage cables are reversed when a $^{63}Ni$ or $^{3}H$ detector is installed.

FIGURE 3-2 REAR POWER PANELS (2700 SERIES)

TABLE 3-2
POWER PANEL

| | |
|---|---|
| ELECT A Signal Cable | Connects input signal from detector A to electrometer channel A. |
| ELECT B Signal Cable | Connects input signal from detector B to electrometer channel B. |
| ELECT A Ignitor Cable | Connects cell and/or ignition voltage from electrometer channel A to detector A. |
| ELECT B Ignitor Cable | Connects cell and/or ignition voltage from electrometer channel B to detector B. |
| RECORDER/INTEGRATOR Output Terminal Strip | Provides terminal for connecting output of Dual/Differential Electrometer or TC Bridge modules to external recorder or integrator. Output signals up to 2.2 V are available at the terminals as indicated below. |

| TARGET (●) INDICATES OUTPUT SIGNAL AVAILABLE FROM TERMINAL STRIP INDICATED | | ELECTROMETER MODE OF OPERATION (SERIES 2740) | | | | | | TC BRIDGE OPERATION (SERIES 2720) |
|---|---|---|---|---|---|---|---|---|
| | | A | B | A-B | B-A | FL-FL | FL-EC | ——— |
| TERMINAL STRIP POSITIONS | ELECT A | ● | ● | ● | ● | ● | ● | ——— |
| | ELECT B | | | | | ● | ● | ——— |
| | TC | | | | | | | ● |

| | |
|---|---|
| Main Power Cable | Connects line power to instrument. |
| F101 TC/ELECT Fuse | Electrometer (2740 Series instruments) or TC Bridge Power Supply (2720 Series instruments) is fused by this 1/4 A fuse. |
| F102 PROG Fuse | Temperature Programmer Module is fused by this 1/2 A fuse (all 2700 Series instruments). |
| F103 TRANS Fuse | Fuse protects line adjusting transformer. For 180 to 265 Vac operation, use 3 A fuse. For 90 to 145 Vac operation use 6 A fuse. |
| F104 MAIN Fuse | Main power to instrument is fused by this 20 A (100 to 130 Vac) or 10 A (200 to 240 Vac) ceramic fuse. |
| CHASSIS Binding Post | Provides connection point for grounding (earth) instrument. |
| COMPUTER Output Terminal Strip | Provides output signals from Electrometer channels A and B and TC Bridge that are unattenuated and are in addition to the outputs available from the RECORDER/INTEGRATOR terminals. |

TABLE 3-2 POWER PANEL (cont'd)

| | |
|---|---|
| Computer Output Terminal Strip (cont'd) | Signal distribution at the COMPUTER output terminal strip is the same as that for the RECORDER/INTEGRATOR output terminal strip.<br><br>Output Capability at ELECT A & B terminals is:<br>— 12V @ 5 ma max, .01 μf to ground without series resistor; .1 μf to ground with 47 ohms in series with output.<br><br>Output Capability at TC terminal is:<br>— Resistive loading influences linearity of output. Negligible error to 50 Kohms. Capacitive loading influences response time; Negligible to .1 μf. |

## 3.4 FLOW CONTROL SYSTEMS

The flow control systems include either a Pressure Gauge module or Rotameter module and a flow control panel. Each of these two modules (set up and operation covered in separate manuals) permit a central location for controlling and monitoring carrier gas flow rate.

The fine metering flow controls located on the left side of the instrument provide a central point for controlling hydrogen and air to each flame ionization (FI) and/or alkali flame ionization (AFID) type detectors. The flow controls are included with the 2740 Series instruments. The hydrogen valve can be used to control purge gas to a $^{3}H$ detector when desired. The purge gas is applied at the HYDROGEN fitting at rear of instrument.

Available as an optional accessory are flow diverters. The flow diverters are mounted adjacent to the fine metering flow controls. These diverters provide a very convenient method for monitoring hydrogen and air flow rate without touching the detectors. The flow diverters are installed when ordered with the instrument; but, they can be ordered separately at a later time and are easily installed.

Located below the flow controls are the gas input connections. CARRIER GAS, HYDROGEN and AIR connections are included on the 2740 Series; only the CARRIER GAS input connections is included on the 2720 Series.

Refer to Figure 3-3 and Table 3-3 for location and description, respectively, of fine metering flow controls.

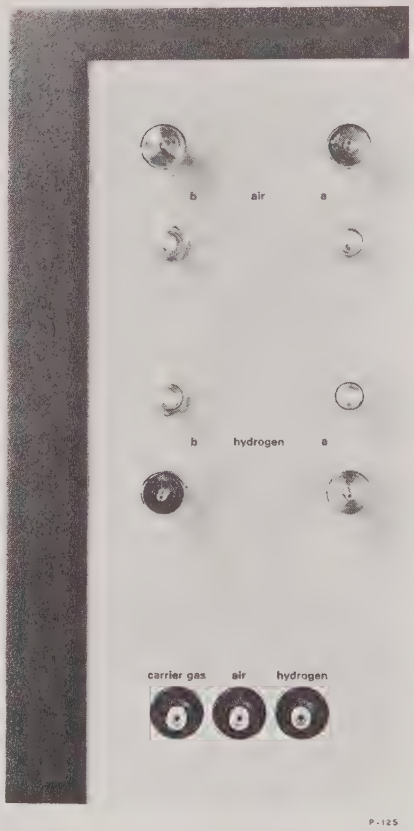



FIGURE 3-3 FLOW CONTROL PANEL

TABLE 3-3
FLOW CONTROL PANEL CONTROLS

| | |
|---|---|
| DETECTOR HYDROGEN A and B Fine Metering Controls | Controls flow of hydrogen to appropriate detector (2740 Series only). Controls must be turned off (Clockwise position) when operating electron capture $^{3}H$ and $^{63}Ni$ detectors. Can be used to control purge gas for electron capture detectors when desired. |
| DETECTOR AIR A and B Fine Metering Controls | Controls flow of air to appropriate detector (2740 Series only). |
| Flow Diverters (optional) | Flow diverters permit monitoring of air or hydrogen of the appropriate flow system (2740 Series only). When diverter is pulled out, gas flow is through associated fine metering valve, diverter, and into detector. With diverter pushed in, gas flow is through fine metering valve and out tube extending from diverter. Be sure to pull diverter out after completion of gas flow measurements. |
| CARRIER GAS Input | For connection of carrier gas line (all 2700 Series instruments). |
| HYDROGEN Input | For connection of hydrogen gas line (2740 Series instruments only). |
| AIR Input | For connection of air line (2740 Series instruments). |

### 3.4.1 Gas Flow Rate Adjustment

Gas flow rates should be adjusted for maximum performance of columns and detectors used. Flow rates should be checked at least once per day and each time a column is installed. Before adjusting flow rates, check the entire flow system for leak(s) as outlined in paragraph 2.5.

Since your instrument can be equipped with a number of various detectors and detector combinations, a general outline for setting up gas flow rates will be discussed here. The details on calibrating the flow control modules and appropriate flow rates for the various detectors are covered in separate manuals.

**• Flow Rate Adjustment when using Thermal Conductivity Detector**

The thermal conductivity detector requires only carrier gas. Adjust gas flow rate as follows:

1) Turn CARRIER GAS FLOW control fully clockwise (OFF).

2) Adjust regulator control on carrier gas tank supply to at least 65 psi or to pressure determined in calibrating the flow control module (Pressure Gauge or Flowmeter) supplied with your instrument.

3) Turn CARRIER GAS FLOW fully counterclockwise (ON).

4) Adjust appropriate flow control valve on flow control module for desired carrier gas flow rate. Refer to separate manuals on Pressure Gauge or Flowmeter module for flowrate calibration records and monitoring procedures.

**• Flow Rate Adjustment when using a Flame Ionization (FID) and/or Alkali Flame Ionization (AFID) Detectors**

The FID and AFID detectors require carrier gas, hydrogen, and air for proper operation. The proper gas mixture ratios and flow rates are discussed in separate FID and AFID Instruction Manuals.

The procedure outlined below applies to both channels A and B using and FID and/or AFID detector combination. Adjust carrier gas, hydrogen, and air flow rates as follows:

— CAUTION —

**When instructed to turn off a fine metering valve, do not overtighten in off position to prevent valve damage.**

*CARRIER GAS*

1) Make sure DETECTOR HYDROGEN A and B controls (located on flow control panel) are turned off (fully clockwise). Do not tighten valves excessively in off position to prevent valve damage.

2) Adjust regulator control on carrier gas tank supply to at least 65 psi or to pressure determined in calibrating the flow control module supplied with your instrument.

3) Turn appropriate A or B flow control valve on flow control module (Pressure Gauge or Flowmeter) for desired carrier gas flow rate. Refer to appropriate manual on Pressure Gauge or Flowmeter module for flow rate calibration records.

*HYDROGEN*

1) Turn on hydrogen source. If you are using a hydrogen generator, allow enough time for generator pressure to build up before proceeding to step (2). If you are using tank hydrogen, set pressure regulator on tank to approximately 10 psi.

2) Adjust DETECTOR HYDROGEN A and/or B control (located on flow control panel) to obtain the desired hydrogen flow through the detector. Monitor hydrogen flow at detector (refer to appropriate detector manual on measuring hydrogen flow rate at detector) with soapbubble flowmeter. Refer to paragraph 3.4.2 for setup and operation of soapbubble flowmeter. You will be measuring the combined flow of carrier gas and hydrogen. Hydrogen flow is equal to the difference between carrier gas flow and total flow rate.

   If your instrument is equipped with flow diverters, simply connect tube from soapbubble flowmeter to exit of appropriate flow diverter, push diverter in as far as it will go, then measure flow and adjust as necessary

with the adjacent DETECTOR HYDROGEN control. Be sure to pull end of diverter out when you finish the flow measurement to prevent hydrogen from being diverted away from the detector.

*AIR*

Air flow measurements can be made in two ways: at the detector base with a standard detector base plug or by optional flow diverters. If you do not have the flow diverters, the detector flame tip or salt tip must be removed and the standard detector base plug installed. The plug blocks off hydrogen and carrier gas but permits the passage of air. One detector base plug is installed on each detector base when the instrument is shipped from the factory (2740 Series only). Refer to appropriate FID and/or AFID Manuals for further information on monitoring air flow at detector(s).

1) Turn on air source. Adjust air flow rate with DETECTOR AIR A and/or B controls to desired flow rate. Monitor air flow rate at plug installed in detector base. Since the soapbubble flowmeter range is not great enough to measure the air flow rate accurately, use any suitable means such as the water displacement method. Be sure to remove plug and reinstall detector before proceeding.

   If your instrument is equipped with flow diverters, simply connect tube from flow measuring device to exit of appropriate flow diverter, push diverter in as far as it will go, then measure flow and adjust as necessary with adjacent DETECTOR AIR control. Be sure to pull end of diverter out when you finish the flow measurement to prevent air from being diverted away from detector.

**• Flow Rate Adjustment when using $^{63}Ni$ and $^{3}H$ Detectors**

Only carrier gas is necessary for operation of the electron capture type detectors. Adjust carrier gas flow rate as follows:

1) Make sure DETECTOR HYDROGEN A and B controls (located on flow control panel) are turned off (fully clockwise). Do not tighten valves excessively in off position to prevent valve damage.

NOTE

**Make sure HYDROGEN input fitting on rear panel is capped off.**

2) Adjust regulator control on carrier gas tank supply to at least 65 psi or to pressure determined in calibrating the flow control module (Pressure Gauge or Rotameter) supplied with your instrument.

3) Turn appropriate A or B flow control valve on flow control module for desired carrier gas flow rate at detector. Refer to appropriate manual on Pressure Gauge or Rotameter module for flow rate calibration records.

### 3.4.2 Soapbubble Flowmeter Operation

To use the soapbubble flowmeter, follow this procedure:

1) Fill rubber reservoir with 2 to 5% soap solution (salt-free detergent such as Liquid Ivory is recommended).

2) Connect tubing to gas outlet to be monitored. Refer to Figure 3-4 for assembly of soapbubble flowmeter.

3) Squeeze reservoir gently until the emerging gas forms ascending soap bubbles in flowmeter.

4) Use a stop watch to measure the time in seconds required for soap bubbles to rise from "0" to "10". Divide this time into 600 to obtain flow rate in ml/min or use chart in Figure 3-5.

## 3.5 PRELIMINARY OPERATING HINTS

The method used in gas chromatography will vary with the type of analysis and the individual techniques of the operator. The following discussion will present some preliminary operating hints that may help in obtaining faster and more accurate results with a gas chromatograph.

### 3.5.1 Columns and Packings

To avoid possible contamination of the detectors, the standard columns supplied with the instrument have been preconditioned. However, all new col-

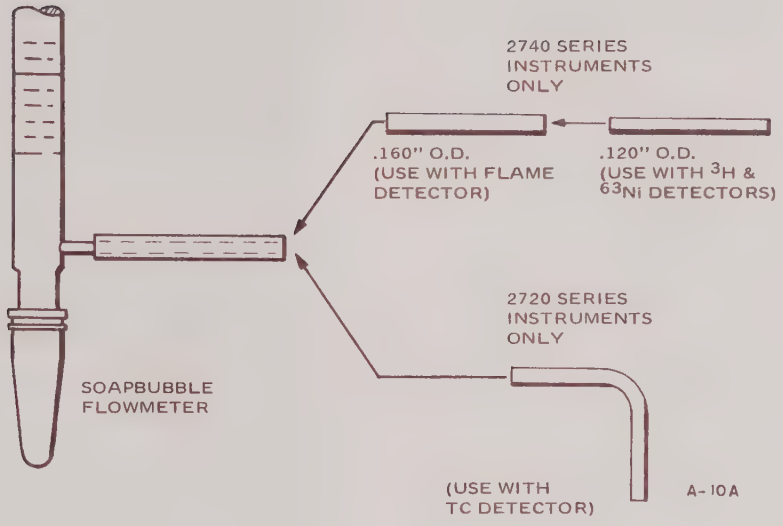



FIGURE 3-4 SOAPBUBBLE FLOWMETER SETUP

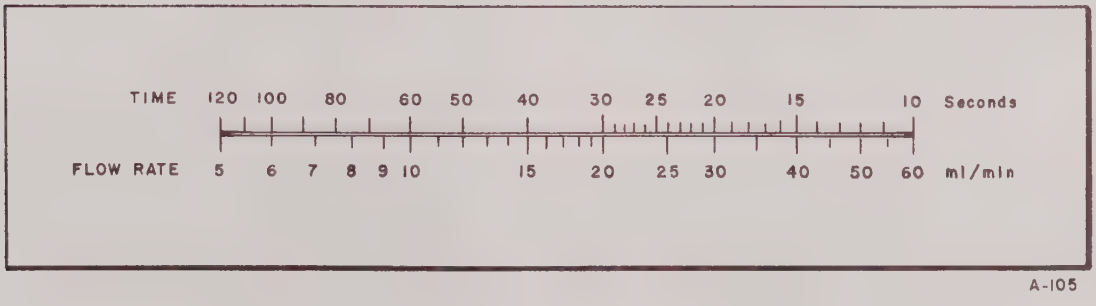



FIGURE 3-5 APPROXIMATION OF FLOW RATE

umns should be preconditioned before being used. Some columns require special preconditioning techniques and these techniques should be followed as required. When no special conditioning techniques are required, precondition columns at about 50°C above the desired operating temperature for 12 hours.

— CAUTION —

**Do not exceed the specified maximum temperature limit of the column's liquid phase being preconditioned.**

Follow this procedure to precondition column:

1) Install columns. Do not connect effluent end of column to crossover fittings at rear of oven.

2) Adjust carrier gas flow rate through columns to 15 to 20 ml/min. Refer to manual on Pressure Gauge or Flowmeter module for procedure on adjusting carrier gas flow rate.

3) On 2720 Series instruments, make sure TC Bridge module is turned off.

4) Set INJECTOR A and B TEMP controls to a low setting. However, the injector temperatures can be adjusted to desired operational temperatures, if desired, since equilibration of injector temperatures require several hours.

5) Set DETECTOR TEMP °C control to desired operational temperature (this will prevent a long delay in operating the instrument after columns have been preconditioned). The detector oven requires from 4 to 5 hours to equilibrate at the desired temperature.

6) Turn on power to instrument.

7) Set COLUMN SAFETY LIMIT °C control to about 25°C above temperature that column oven will be operated during analysis.

8) Adjust temperature programmer module so that column-oven temperature is approximately 50°C higher than the desired operating temperature for the column used. However, do not exceed maximum operating temperature of column packing material. Refer to appropriate instruction manual concerning the operation of the temperature programmer.

9) Set COLUMN OVEN switch to COLUMN & FAN position.

10) Monitor column-oven temperature with pyrometer and readjust temperature as required.

11) Bake columns for at least 12 hours with carrier gas flowing as set in step (2).

NOTE

**If you prefer to raise the column-oven temperature slowly when conditioning a column, use temperature programmer with slow programming rates.**

### 3.5.2 Carrier Gas Flow Rates

The flow rate of carrier gas through the instrument will affect sample retention time and the amplitude of the recorded peaks. Flow rates between 15 and 30 ml/min should offer the best compromise between sensitivity and resolution with 1/8-inch OD columns. Flow rates of 40 to 100 ml/min are recommended for 1/4-inch OD columns.

### 3.5.3 Detector Temperatures

The detector temperature should be held approximately 25 to 50°C higher than the maximum operating column-oven temperature. This will prevent condensation of sample components in the detector. Because the detector-oven temperature requires approximately four hours to equilibrate, its temperature should be changed only when necessary.

NOTE

**A thermal switch is automatically placed in the detector heater circuit when an electron capture ($^3H$) detector is installed. The thermal switch automatically prevents the detector foil temperature from exceeding 225°C.**

### 3.5.4 Injector Temperatures

Injector temperatures should be set approximately 25 to 50°C above the highest column-oven temperature. If thermal decomposition results when the sample is injected into the heated injector, on-column injection should be used. To inject a sam-

ple directly into the column, a needle of at least 2 inches in length must be used.

### 3.5.5 Isothermal Operation

Generally, the column-oven temperature is set about 40°C below the boiling point of the highest boiling compound in the sample. Depending on the sample, you may improve results by increasing or decreasing the oven temperature. As a general rule, retention time decreases about 3% for every one degree C increase in temperature.

### 3.5.6 Programmed Operation

Programmed temperature techniques permit the column oven to operate through a temperature range to elute the lowest boiling compounds in a well-spaced pattern and the highest boiling compounds in a reasonable period of time.

### 3.5.7 Sample Injection

Prepare syringe by flushing several times with the sample before filling. Solid samples can be dissolved in a solvent having a lower or higher boiling point than the sample's components. This will allow the solvent peak to elute before or following the sample's components.

Good sample injection techniques are learned with practice. The syringe is held perpendicular to the injector septum to ensure reproducibility. Sample injection must be in a smooth but rapid motion to permit complete vaporization of the sample. This technique will prevent peak tailing due to poor injection. For further discussion, refer to *Syringe Handling Techniques* at end of this manual.

### 3.5.8 Preliminary Analysis

The results of a preliminary analysis will aid the operator in determining optimum operating conditions such as sample sizes, temperature parameters, flow rates, and attenuation. If the initial (or isothermal) temperature is too low, the first peaks will be well spaced, but the remaining peaks will be too widely spaced. If the initial temperature is too high, the first peaks will be too closely spaced or overlapped. Therefore, programmed temperature techniques may offer the best solution for obtaining satisfactorily spaced peaks.

### 3.5.9 Carrier Flow Balancing for Dual-Column Differential Operation

The primary advantage of using dual columns is to minimize the effects of liquid phase bleeding from the columns during temperature programming. Liquid phase bleeding occurs when the liquid phase vapor concentration in the carrier gas becomes high enough to cause drifting of the chromatographic baseline. The rate of liquid phase bleeding increases as the column temperature increases. The following procedures will minimize baseline drift resulting from liquid phase bleeding.

- **Flow Balancing for Thermal Conductivity Detector* (2720 Series)**

Proceed as follows:

1) Adjust carrier gas flow rate through matched reference and analytical columns to desired value. Measure flow rate at collector exit tubes. Refer to appropriate manual on Pressure Gauge or Rotameter module for procedure on adjusting carrier gas flow rate. Balance TC Bridge Power Unit as outlined in appropriate manual. When balancing the TC Bridge Power Unit/Detector, use an attenuator setting lower than you plan to use during normal operation.

2) Heat column oven to 15°C above the desired oven temperature program limit. Make sure flow rate in analytical column has not changed. Refer to instruction manual on column-oven temperature programmer module.

3) If baseline drift occurs, compensate by adjusting reference column flow rate to return recorder pen to original baseline.

NOTE

**The effects of flow rate changes occur slowly at higher temperatures; therefore, allow enough time for flow system to equilibrate — up to 5 minutes might be necessary.**

4) Lower column-oven temperature to lower program limit.

5) If baseline drift occurs, adjust FINE BALANCE control on TC Bridge module to return recorder pen to original baseline. Again, allow time for flow system to equilibrate.

---

**Columns must be preconditioned (see paragraph 3.5.1). Matched columns must be preconditioned at the same temperature and flow rate.*

6) Repeat balancing procedure by adjusting reference column flow rate at high temperature and FINE ZERO at low temperature until baseline drift is minimized.

• **Flow Balancing for Dual Flame Ionization Detectors* (2740 Series)**
Proceed as follows:

1) Balance Electrometer as outlined in instruction manual on Electrometer module. Use an attenuator setting lower than you plan to use during normal operation.

2) Adjust flow rate of matched columns to approximately 25 ml/min for 1/8-inch columns. Refer to instruction manual on Pressure Gauge or Flowmeter module for instructions on adjusting carrier gas flow rate.

3) Ignite flames and turn ionization voltage on (CELL VOLTAGE switches in ON position). Refer to instruction manual on Electrometer module.

4) Adjust column oven to desired initial temperature. Refer to instruction manual on column-oven temperature programmer module.

5) Heat column oven to 15°C above final operating program temperature.

6) If baseline drift occurs, compensate by adjusting the reference column (column B) carrier gas flow rate to return recorder pen to original baseline. Column A is used as the analytical side.

NOTE

**The effects of flow rate changes occur slowly at higher temperatures; therefore, allow enough time for flow system to equilibrate — up to 5 minutes might be necessary.**

7) Cool column oven to initial program starting temperature. Allow flow system to equilibrate.

8) If the baseline has shifted, compensate by adjusting BUCKING control on Electrometer module (reference channel) to return recorder pen to original baseline.

9) Repeat above procedure (5) through (8) to make sure column bleed effects have been minimized.

## 3.6 OPERATION WITH THERMAL CONDUCTIVITY DETECTION (2720 Series)

Outlined below is a condensed procedure on operating your instrument with a thermal conductivity detector. Where more detailed setup and operational information is required for a particular module, refer to the appropriate manual included with this basic instrument manual. All other controls discussed can be found on the main control or rear power panels of the basic instrument.

Before operating your instrument, make sure that all installation and leak checks have been completed as outlined in Section 2.

Operate instrument as follows:

1) Adjust carrier gas flow as outlined in paragraph 3.4.1.

2) Turn on MAIN POWER switch on main control panel. Light above switch should light indicating power is on.

— CAUTION —

**To prevent damage to TC filaments, do not turn on TC Bridge Module or apply current to TC filaments unless carrier gas is flowing through TC detector.**

3) Set COLUMN SAFETY LIMIT °C control to about 25°C higher than desired column-oven temperature.

4) Turn on DETECTOR TEMP and INJECTOR A and B TEMP switches. Lights above switches should turn on indicating power is on.

5) Turn DETECTOR TEMP °C control to desired setting. Allow at least 4 to 5 hours for detector-oven temperature to equilibrate.

6) Adjust INJECTOR A and B TEMP controls for desired injector temperature. Refer to Figure 3-6 for plot of injector temperature vs control position.

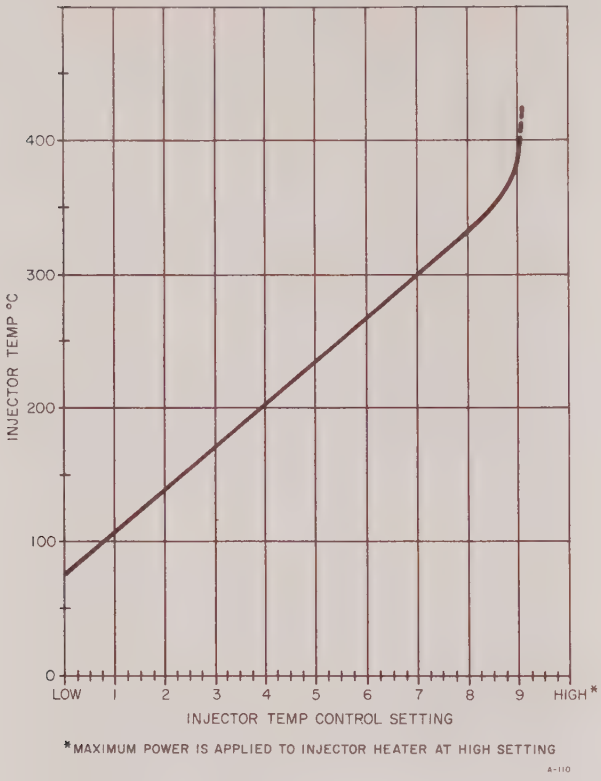



**FIGURE 3-6 INJECTOR TEMPERATURE CURVE**

NOTE

**Monitor detector and injector temperatures on pyrometer located on main control panel.**

7) Turn on temperature programmer module and select desired initial column-oven temperature. Refer to manual on temperature programmer module being used for setup and operational procedures.

8) Set COLUMN OVEN switch on main control panel to OVEN & FAN position. Light above switch should turn on indicating power is on.

9) Turn on strip-chart recorder and set up as required.

10) Set POLARITY switch (located on TC Bridge Module) to either the + or - position to keep peaks on scale.

11) Adjust TC Bridge Module for desired operating parameters. Refer to manual on TC Bridge Module.

12) Make sure all thermal regions are at the proper temperature (via front panel pyrometer), i.e., injector, detector, column oven, then inject sample into appropriate injector (usually B injector). For programmed operation, start program then immediately inject sample.

13) Observe chromatogram and adjust ATTENUATOR control on TC Bridge Module for best peak resolution. Note ATTENUATOR settings on chromatogram.

## 3.7 OPERATION WITH IONIZATION DETECTION (2740 Series)

Outlined below is a condensed procedure on operating your instrument with ionization detectors, e.g., FID, AFID, $^{3}H$, and $^{63}Ni$. Where more detailed information is required for setup and operation of a module or detector, refer to the appropriate manual included with this basic instrument manual. All other controls discussed can be found on the main control or rear power panels of the basic instrument.

Installation and leak checks should be completed as outlined in Section 2 before operating the instruments. Setup and operational information on each detector is covered in separate manuals. Precautions that must be observed when operating a particular detector or detector combinations are also covered in these manuals.

Whenever a $^{3}H$ detector is installed, the detector-oven temperature is limited to prevent the detector foil temperature from exceeding 225°C. This prevents the release of $^{3}H$ from the foil. The temperature is limited by a thermal switch which is automatically placed in the detector heater circuit with the installation of the $^{3}H$ detector. A microswitch which enables the thermal switch is located directly under the bottom probe of the detector tower. A large band around the probe engages the switch when the tower is installed, thereby activating the microswitch.

Operate instrument as follows:

1) Adjust carrier gas, hydrogen, and air flow rates for the FID and/or AFID detector(s) as outlined in the appropriate detector manual. For $^{3}H$ and $^{63}Ni$ detectors, only carrier gas is required. Refer to $^{3}H$ and $^{63}Ni$ manuals for flow rate information.

NOTE

**Make sure to turn off DETECTOR AIR AND $H_2$ valves on flow control panel for channel(s) incorporating the $^{3}H$ and/or $^{63}Ni$ detector(s).**

2) Turn on MAIN POWER switch on main control panel. Light above switch should light indicating power is on.

3) Set COLUMN SAFETY LIMIT °C control to about 25°C higher than desired column-oven temperature.

4) Turn on DETECTOR TEMP and INJECTOR A and/or B switches. Lights above switches should turn on indicating power is on.

5) Turn DETECTOR TEMP °C control to desired setting. Allow at least 4 to 5 hours for detector-oven temperature to equilibrate.

6) Adjust INJECTOR A and/or B TEMP controls for desired injector temperature. Refer to Figure 3-6 for plot of injector temperature vs control position.

NOTE

**Monitor detector and injector temperatures on pyrometer located on main control panel.**

7) Turn on temperature programmer module and select desired initial column-oven temperature. Refer to manual on temperature programmer module being used for setup and operational procedures.

8) Set COLUMN OVEN switch on main control panel to OVEN & FAN position. Light above switch should turn on indicating power is on.

9) Turn on strip-chart recorder and set up as required.

10) Adjust Electrometer Module for desired operating parameters with detector or detector combinations being used. Refer to manual on Electrometer module.

11) Make sure all thermal regions are at the proper temperature (via front panel pyrometer), i.e., injector, detector, column oven, then inject sample into appropriate injector. For programmed operation, start column-oven temperature programmer then immediately inject sample.

12) Observe chromatogram and adjust controls on Electrometer module as required. Note control settings on chromatogram.

# SECTION 4
# SERVICING

## 4.1 GENERAL

This section of the manual discusses routine maintenance and cleaning procedures, provides a comprehensive table of chromatographic interpretations including operational problems and suggested remedies, and a calibration procedure for the COLUMN SAFETY LIMIT °C control.

## 4.2 MAINTENANCE AND CLEANING

Sometimes column materials leak into the detector base causing the internal parts of the bases and crossover lines to become contaminated. Therefore, it may be necessary to clean the detector bases occassionally. Both detector bases and crossover lines are cleaned in the same manner. Clean detector bases and crossover lines as follows:

NOTE

**If detector base is contaminated and requires cleaning, the detector components should also be cleaned. Refer to appropriate detector manual.**

1) Turn off cell voltages at Electrometer.

WARNING

**To avoid a shock hazard, make sure detector cell voltage is turned off at Electrometer and that the cell voltage cable is disconnected from the tower probe.**

2) Remove any ionization detectors installed.

3) Remove columns.

4) Connect a length (12 to 14 inches) of clean 1/8-inch S.S. tubing between injector and crossover fitting of system to be cleaned. A dummy column can also be used.

5) It is necessary to apply some positive pressure to the air and hydrogen lines to prevent cleaning solvent from backing into lines. Normal air flow rates applied at AIR input fitting and a few ml/min of some inert gas, e.g., nitrogen, applied at $H_2$ input fitting is adequate. Make sure AIR and $H_2$ FINE METERING controls are turned on (counterclockwise).

6) Replace insulation pad and cover on detector oven, then heat detector oven to approximately 150°C.

7) Set injector and column-oven temperature to 150°C.

8) Adjust carrier gas flow rate of system being cleaned to at least 10 ml/min.

9) Make six 50 µl injections of distilled water at three minute intervals.

10) Repeat step (9) using acetone.

11) Clean second detector base and crossover line, if necessary, at this time.

12) Increase detector base temperature to 200°C. Dry internal components of detector base and crossover line by purging with nitrogen. Since two to four hours are required for equilibration of detector base temperature, you may wish to adjust detector base temperatures to actual operating condition during the purging process. This will save you time in placing the instrument into operation.

13) Remove gas line from $H_2$ fitting and reconnect hydrogen line.

14) Remove tubing (or dummy columns) installed in oven.

15) Reinstall detector(s) on base.

## 4.3 CHROMATOGRAPHIC INTERPRETATIONS

Table 4-1 lists many problems which you might encounter in operating your instrument. Included with each problem is a possible cause and remedy. The problems are illustrated with chromatograms which are typical of the problems discussed. Refer to Table 4-1.

TABLE 4-1
CHROMATOGRAPHIC INTERPRETATIONS

| Symptom | Possible Cause | Checks and/or Remedy |
|---|---|---|
| 1. No peaks.<br> <br><br>NOTES:<br>1. Injection point same for all chromatograms.<br>2. Time is shown as indicated by arrow on all chromatograms. | 1a. Bridge power supply or electrometer power off.<br>1b. No carrier gas flow.<br>1c. Recorder improperly connected.<br>1d. Recorder defective.<br>1e. Injector temperature too cold. Sample not being vaporized.<br>1f. Hypodermic syringe leaking or plugged.<br>1g. Injector septum leaking.<br>1h. Column connections loose.<br>1i. Flame out (FID only)<br>1j. No cell voltage being applied to detector (all ionization detectors). | 1a. Turn power on. Check electrometer or bridge power supply fuse.<br>1b. Turn carrier gas flow on and adjust to proper setting. If carrier lines are obstructed, remove obstruction. Replace carrier gas tank if empty.<br>1c. Check recorder connections. Remove any jumper lines connecting either recorder input connection to ground or shield.<br>1d. See recorder instruction manual for troubleshooting procedures.<br>1e. Increase injector temperature.<br>1f. Replace syringe.<br>1g. Replace injector septum.<br>1h. Tighten column connections.<br>1i. Light flame.<br>1j. Turn on cell voltage. Also, check for defective detector cables. |
| 2. Poor sensitivity with normal retention time.<br> | 2a. Attenuation too high.<br>2b. Insufficient sample size.<br>2c. Poor sample injection technique.<br>2d. Syringe leaking or plugged.<br>2e. Carrier gas leak. (Particularly at injector septum).<br>2f. Detector bridge current too low (TC detector only).<br>2g. Detector cell voltage off (FID only). | 2a. Reduce attenuation. Lower electrometer sensitivity.<br>2b. Increase sample size.<br>2c. Review sample injection techniques. See Syringe Handling Techniques at back of manual.<br>2d. Replace syringe.<br>2e. Correct leak.<br>2f. Increase detector current.<br>2g. Turn on cell voltage. |

TABLE 4-1 CHROMATOGRAPHIC INTERPRETATIONS (cont'd)

| Symptom | Possible Cause | Checks and/or Remedy |
|---|---|---|
| 3. Poor sensitivity with increased retention time. | 3a. Carrier gas flow rate too low.<br>3b. Flow leaks downstream of injector.<br>3c. Injector septum leaking. | 3a. Increase carrier gas flow rate. If carrier gas lines are obstructed, locate and remove obstruction.<br>3b. Locate leak and correct.<br>3c. Replace injector septum. |
| 4. Negative peaks. | 4a. Recorder improperly connected. Input leads reversed.<br>4b. Sample injected in wrong column.<br>4c. MODE selector in wrong position (differential mode). | 4a. Check recorder connections. Reverse leads.<br>4b. Inject sample in proper column.<br>4c. Ensure MODE selector is in correct position for column being used as analytical column. |
| 5. Irregular baseline drift when operating isothermally. | 5a. Poor instrument location.<br>5b. Instrument not properly grounded.<br>5c. Column packing bleeding.<br>5d. Carrier gas leak.<br>5e. Crossover lines to detector oven contaminated.<br>5f. Detector block contaminated (TC detector).<br>5g. Detector base contaminated (Ionization detectors).<br>5h. Detector-oven temperature not stable (TC detector). | 5a. Move instrument to a different location. Instrument should not be placed directly under heater or air conditioner blower, or any other place where it is subject to excessive drafts and ambient temperature changes.<br>5b. Make sure instrument and recorder are connected to good earth ground.<br>5c. Recondition column. Some columns are impossible to condition well at the desired operating conditions. These columns will always produce some baseline drift, particularly when operating at high sensitivity conditions.<br>5d. Locate leak and correct.<br>5e. Clean crossover lines.<br>5f. Clean detector block.<br>5g. Clean detector base and detector.<br>5h. Make sure insulation plugs are installed in oven lid if ionization detectors are not installed; also, make sure there are no gaps in the detector-oven insulation which will allow room air to enter detector oven. |

TABLE 4-1 CHROMATOGRAPHIC INTERPRETATIONS (cont'd)

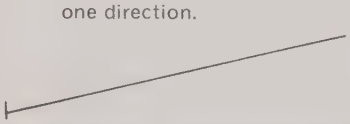
| Symptom | Possible Cause | Checks and/or Remedy |
|---|---|---|
| | 5i. Poor carrier gas regulation. | 5i. Check carrier gas regulator and flow controllers to ensure proper operation. Make sure carrier gas tank has sufficient pressure. |
| | 5j. Poor $H_2$ or air regulation (FID only). | 5j. Check $H_2$ and air flow to ensure proper flow rate and regulation. |
| | 5k. Detector filaments defective (TC detector only). | 5k. Replace TC detector assembly. |
| | 5l. Electrometer defective (Ionization detectors). | 5l. Consult nearest Varian Aerograph representative on troubleshooting electrometer. |
| | 5m. Recorder defective. | 5m. Disconnect input leads to recorder and short the recorder input with a piece of wire. If drift continues, recorder is defective. See recorder manual. Especially check recorder gain adjustment, damping adjustment, ground connection, and amplifier. |
| | 5n. TC Power Supply defective. | 5n. Replace Power Supply. |
| 6. Sinusoidal baseline drift. | 6a. Detector oven insulation faulty. | 6a. Make sure insulation plugs are installed in oven lid if flame detectors are not installed; also make sure oven lid is securely in place. |
| | 6b. Detector-oven temperature controller defective. | 6b. Replace detector-oven temperature controller, and/or temperature-sensing probe. |
| | 6c. Column oven temperature controller defective. | 6c. Replace column-oven temperature control module and/or temperature-sensing probe. |
| | 6d. Column oven temperature limit control set too low. | 6d. Set column oven temperature limit control to higher setting. Must be set higher than highest desired operating temperature of the column oven. |
| | 6e. Carrier gas flow regulator defective. | 6e. Replace carrier gas flow regulator. |
| | 6f. Carrier gas tank pressure too low to allow regulator to control properly. | 6f. Replace carrier gas tank. |
| 7. Constant baseline drift in one direction.  | 7a. Detector temperature increasing (decreasing) | 7a. Allow sufficient time for detector to stabilize after changing its temperature. Particularly important with TC detector. Detector block will lag the indicated temperature somewhat because of its large mass. |

TABLE 4-1 CHROMATOGRAPHIC INTERPRETATIONS (cont'd)

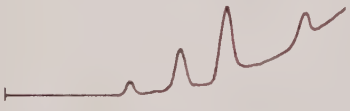
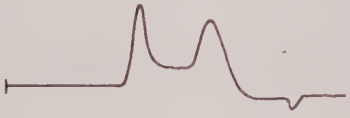
| Symptom | Possible Cause | Checks and/or Remedy |
|---|---|---|
| | 7b. Flow leak down stream of column effluent end (TC detector only). | 7b. A very small diffusion leak will allow a small amount of air to enter the detector at a constant rate. This in turn will oxidize the affected elements at a constant rate while slowly changing their resistance. Locate leak and correct. These are often very slight leaks, and difficult to find. Use high carrier gas pressure (60-70 psig) if necessary. |
| | 7c. Defective detector filaments (TC detector). | 7c. Replace detector. |
| | 7d. Defective power supply (TC detector). | 7d. Replace power supply. |
| | 7e. Defective electrometer (Ionization detectors). | 7e. Consult nearest Varian Aerograph representative for troubleshooting electrometer. |
| 8. Rising baseline when temperature programming.  | 8a. Increase in column "bleed" when temperature rises. | 8a. Use reference column (dual column operation) and condition column. |
| | 8b. Column flow rates not balanced for optimum performance. | 8b. Balance column flow rates. |
| | 8c. Column contaminated. | 8c. Recondition column. |
| 9. Irregular baseline shifting  | 9a. Excessive column "bleeding" from well conditioned columns. | 9a. Use different columns. Some packing materials are highly volatile so that they cannot be temperature programmed without difficulty. This will occur even on well conditioned columns where the carrier gas flow rates have been carefully optimized. |
| | 9b. Column not properly conditioned. | 9b. Condition column. |
| | 9c. Carrier gas flow rates not balanced for optimum performance. | 9c. Balance column flow rates. |
| | 9d. Column contaminated. | 9d. Recondition column. |

TABLE 4-1 CHROMATOGRAPHIC INTERPRETATIONS (cont'd)

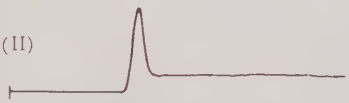
| Symptom | Possible Cause | Checks and/or Remedy |
|---|---|---|
| 10. Baseline "stepping". Baseline does not return to zero, attenuation is incorrect, and peaks are "flat topped". Recorder pen easily moved upscale or downscale with finger pressure.<br>(I)<br> <br>(II)<br>  | 10a. Recorder gain and/or damping control improperly adjusted.<br>10b. Instrument and/or recorder not properly grounded.<br>10c. Low level ac signal being fed to recorder. | 10a. Adjust recorder gain and/or damping control. Refer to recorder instruction manual. When adjusted properly recorder pen cannot be moved easily with finger pressure.<br>10b. Make sure instrument and recorder are connected to good earth ground.<br>10c. Install filter capacitor (about 0.25 uf 150 Vdc) from either + or - input of recorder to ground. Determine by trial and error which position (or both works best. Do not connect capacitor across recorder input (i.e., from + to - connection). |
| 11. Baseline cannot be zeroed. | 11a. Adjustable zero on recorder improperly set.<br>11b. Detector filaments out of balance (TC detector).<br>11c. Defective power supply (TC detector).<br>11d. Excessive signal from column "bleed" (especially FID).<br>11e. Dirty detector (FID and EC)<br>11f. Defective electrometer.<br>11g. Recorder improperly connected.<br>11h. Recorder defective. | 11a. Short recorder input with a piece of wire and adjust zero. See recorder instruction manual.<br>11b. Adjust filament center balance. Replace detector if necessary.<br>11c. Replace power supply.<br>11d. Use different column with less "bleed".<br>11e. Clean detector base and detector.<br>11f. Consult nearest Varian Aerograph representative for troubleshooting electrometer.<br>11g. Check recorder connections. Remove any jumper lines connecting either recorder input connection to ground or shield.<br>11h. See recorder instruction manual. |
| 12. Sharp "spiking" at irregular intervals.<br> | 12a. Quick atmospheric pressure changes from opening and closing doors, blowers, etc.<br>12b. Dust particles or other foreign material burned in flame (FID only). | 12a. Relocate instrument to minimize problem. Also do not locate under heater or air conditioner blowers.<br>12b. Keep detector chamber free of insulation, molecular sieve (from air filter), dust particles, etc. Blow out or vacuum detector to remove dust. |

TABLE 4-1 CHROMATOGRAPHIC INTERPRETATIONS (cont'd)

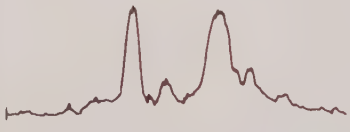
| Symptom | Possible Cause | Checks and/or Remedy |
|---|---|---|
| | 12c. Dirty insulators and/or connectors (Ionization detectors). | 12c. Clean insulators and connectors with residue free solvent. Do not touch with bare fingers after cleaning. |
| | 12d. Power supply defective (TC detector). | 12d. Replace power supply. |
| | 12e. Defective electrometer (Ionization detectors). | 12e. Consult nearest Varian Aerograph representative for troubleshooting electrometer. |
| 13. Short spikes or peaks at regular intervals. | 13a. Condensation in flow lines causing carrier gas to bubble through. | 13a. Heat lines to remove condensation or blow out lines. |
| | 13b. Bubble meter with high liquid level attached to detector exit line (TC detector). | 13b. Remove soapbubble flowmeter tube from exit line. |
| | 13c. Water condensation in hydrogen line coming from Hydrogen Generator (FID only). | 13c. Remove water from line and replace hydrogen filter. |
| 14. High background signal (noise).  | 14a. Contaminated column or excessive bleed from column. | 14a. Recondition column. |
| | 14b. Contaminated carrier gas. | 14b. Replace or regenerate carrier gas filter. Regenerate filter by heating to about 175-200°C and purging overnight with dry nitrogen. |
| | 14c. Carrier gas flow rate too high. | 14c. Reduce carrier gas flow rate. |
| | 14d. Carrier gas flow leak. | 14d. Locate leak and correct. |
| | 14e. Loose connections. | 14e. Make sure all interconnecting plug and screw connections are tight. Make sure modules are properly seated in their plug-in connectors. |
| | 14f. Bad ground connection. | 14f. Ensure all ground connections are tight and connected to a good earth ground. |
| | 14g. Dirty switches. | 14g. Locate dirty switch; spray with a contact cleaner and rotate switch through its positions several times. |
| | 14h. Dirty recorder slidewire. | 14h. Clean recorder slidewire. See recorder manual. If slide wire is dirty, noise will always appear at same area or areas on chart and noise level will generally remain constant regardless of attenuation. |

TABLE 4-1 CHROMATOGRAPHIC INTERPRETATIONS (cont'd)

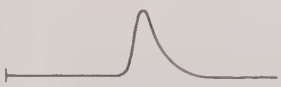
| Symptom | Possible Cause | Checks and/or Remedy |
|---|---|---|
| | 14i. Defective recorder. | 14i. Short recorder input with piece of wire. If noise continues, check recorder. See recorder manual. |
| | 14j. Dirty injector. | 14j. Clean injector tube and replace septum. |
| | 14k. Dirty crossover lines from column to detector oven. | 14k. Clean crossover lines. |
| | 14l. Dirty detector (TC detector). | 14l. Clean detector block. |
| | 14m. Defective detector filaments (TC detector). | 14m. Replace detector assembly. |
| | 14n. Defective bridge circuitry and/or power supply (TC detector). | 14n. Replace TC module. |
| | 14o. Hydrogen flow rate too high or too low (FID detector). | 14o. Adjust hydrogen flow rate to proper level. |
| | 14p. Air flow rate too high or too low (FID detector). | 14p. Adjust air flow rate to proper level. |
| | 14q. Air or hydrogen contaminated (FID detector). | 14q. Replace or regenerate air and hydrogen filters. |
| | 14r. Water condensing inside flame detector shell (FID detector). | 14r. Raise detector temperature to approximately 100°C to eliminate condensation. |
| | 14s. Defective detector cables (Ionization detectors). | 14s. Replace detector cables. |
| | 14t. Dirty detector insulators (Ionization detectors). | 14t. Clean insulators thoroughly with residue free solvent. Do not touch clean insulators with fingers. |
| | 14u. Dirty detector base and/or tower (Ionization detectors). | 14u. Clean detector base and tower. Use ultrasonic bath if possible. |
| 15. Tailing peaks.  | 15a. Injector temperature too high or too low. | 15a. Readjust injector temperature. |
| | 15b. Injector tube dirty (sample or septum residue). | 15b. Clean injector tube with solvent and pipe cleaner. |
| | 15c. Column-oven temperature too low. | 15c. Increase column-oven temperature. Do not exceed recommended maximum temperature for packing material. |
| | 15d. Poor sample injection technique. | 15d. See Syringe Handling Techniques at back of manual. |

TABLE 4-1 CHROMATOGRAPHIC INTERPRETATIONS (cont'd)

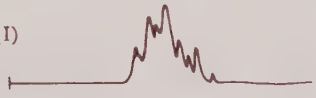
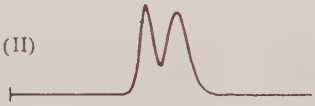
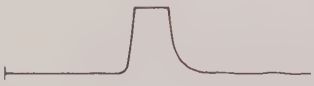
| Symptom | Possible Cause | Checks and/or Remedy |
|---|---|---|
| | 15e. Wrong column. Interaction between sample material and column solid support and/or liquid phase. | 15e. Use different column. Consult nearest Varian Aerograph representative for column advice. |
| 16. Leading peaks. | 16a. Column overloaded. Sample size too large for column diameter and length.<br>16b. Sample condensed in system. | 16a. Decrease sample size.<br>16b. Ensure injector and detector temperatures are correct. |
| 17. Unresolved peaks. (I)  (II)  | 17a. Column oven temperature too high.<br>17b. Column too short.<br>17c. Liquid phase has "baked off" column support material..<br>17d. Wrong column. Incorrect choice of liquid phase and/or solid support.<br>17e. Carrier gas flow rate too high.<br>17f. Poor injection techniques. | 17a. Lower column oven temperature.<br>17b. Use longer column.<br>17c. Replace column.<br>17d. Use different column. Consult nearest Varian Aerograph representative for column advice.<br>17e. Reduce carrier gas flow rate.<br>17f. See Syringe Handling Techniques at back of manual. |
| 18. Round top peaks. | 18a. Operating beyond linear dynamic range of detector output (especially noticeable with EC detector).<br>18b. Recorder gain too low. | 18a. Reduce sample size.<br>18b. Adjust recorder gain to proper setting. See recorder manual. |
| 19. Square top peaks.  | 19a. Electrometer input saturated (Ionization detectors).<br>19b. Recorder slide wire defective or mechanical binding of drive system. | 19a. Reduce sample size. Decrease electrometer sensitivity via RANGE selector.<br>19b. Check recorder operation on a separate instrument or millivolt source. See recorder manual. |

TABLE 4-1 CHROMATOGRAPHIC INTERPRETATIONS (cont'd)

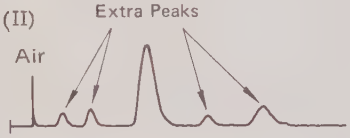
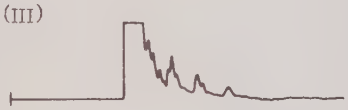
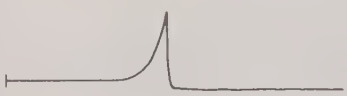
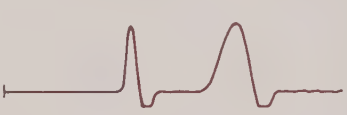
| Symptom | Possible Cause | Checks and/or Remedy |
|---|---|---|
| 20. Extra peaks.<br>(I)<br> <br>(II) Extra Peaks<br>Air<br>  <br>(III)<br>  | 20a. Heavy residual material being eluted from previous sample injection (chromatogram I).<br>20b. Moisture or other impurities from carrier gas condensed on cool column head are eluted during temperature programmed run (chromatogram I).<br>20c. Air peak (chromatogram II).<br>20d. "Ghost peaks". Desorption from column packing when solvent injected (chromatogram II).<br>20e. Sample decomposition (chromatogram II and III).<br>20f. Dirty sample (chromatogram III).<br>20g. Sample interaction with liquid phase or solid support of column packing (chromatograms I, II, and III).<br>20h. Contamination from glassware, syringes, etc. (chromatograms I, II, and III). | 20a. Allow sufficient time for previous sample injections to be eluted.<br>20b. Install, replace, or regenerate carrier gas filter. This is particularly a problem when using a TC detector.<br>20c. Normal when making syringe injections.<br>20d. Make several solvent injections and recondition column.<br>20e. Lower injector temperature. Use different column if packing material causing or catalyzing decomposition.<br>20f. Ensure proper clean-up of sample prior to injection.<br>20g. Use different column. Consult nearest Varian Aerograph representative for column advice.<br>20h. Make sure glassware, syringes, etc., are clean. |
| 21. Sudden drop-off of otherwise normal peak. Recorder pen returns to point below previous baseline. Flame becomes extinguished (FID detector).<br>  | 21a. Sample size too large.<br>21b. Sample contains greater $O_2$ content than combustion air, causing flash back.<br>21c. Loss of hydrogen or air.<br>21d. Carrier gas flow rate too high.<br>21e. Flame tip fouled.<br>21f. Hydrogen generator "kick-off" due to back pressure surge. | 21a. Reduce sample size.<br>21b. Dilute sample with inert gas or use oxygen rather than air to support flame combustion.<br>21c. Reestablish proper air and hydrogen flow rates.<br>21d. Establish proper carrier gas flow rate.<br>21e. Clean or replace flame tip.<br>21f. Reset hydrogen generator. If it "kicks-off" again look for obstruction in hydrogen flow system. Clean $H_2$ flow system and reset generator. Check hydrogen generator filters. |

TABLE 4-1 CHROMATOGRAPHIC INTERPRETATIONS (cont'd)

| Symptom | Possible Cause | Checks and/or Remedy |
|---|---|---|
| 22. Negative dips after peaks (EC detector).  | 22a. Contaminated detector. | 22a. Clean detector. |
| 23. "Screw cap effect" with EC detector. Large broad tailing peak. | 23a. Sample container cap liner partially dissolved in sample solvent. | 23a. Line sample container cap with metal foil, or use glass or polyethylene stoppered container. |

## 4.4 CALIBRATION PROCEDURES

If the COLUMN SAFETY LIMIT °C control is found to be out of calibration, recalibrate as follows:

1) Set COLUMN SAFETY LIMIT °C control fully clockwise.

2) Adjust column-oven temperature to 250°C.

3) Adjust COLUMN SAFETY LIMIT °C control counterclockwise until control indicates 250°C. Power to column oven should turn off at 250°C. If power is not cut off to the column oven, adjust control to point where power is cut off to column oven. Then, loosen set screws securing control knob and rotate knob to 250°C position. Retighten set screws securing knob.

# SECTION 5
# ILLUSTRATED PARTS LISTS/SCHEMATICS

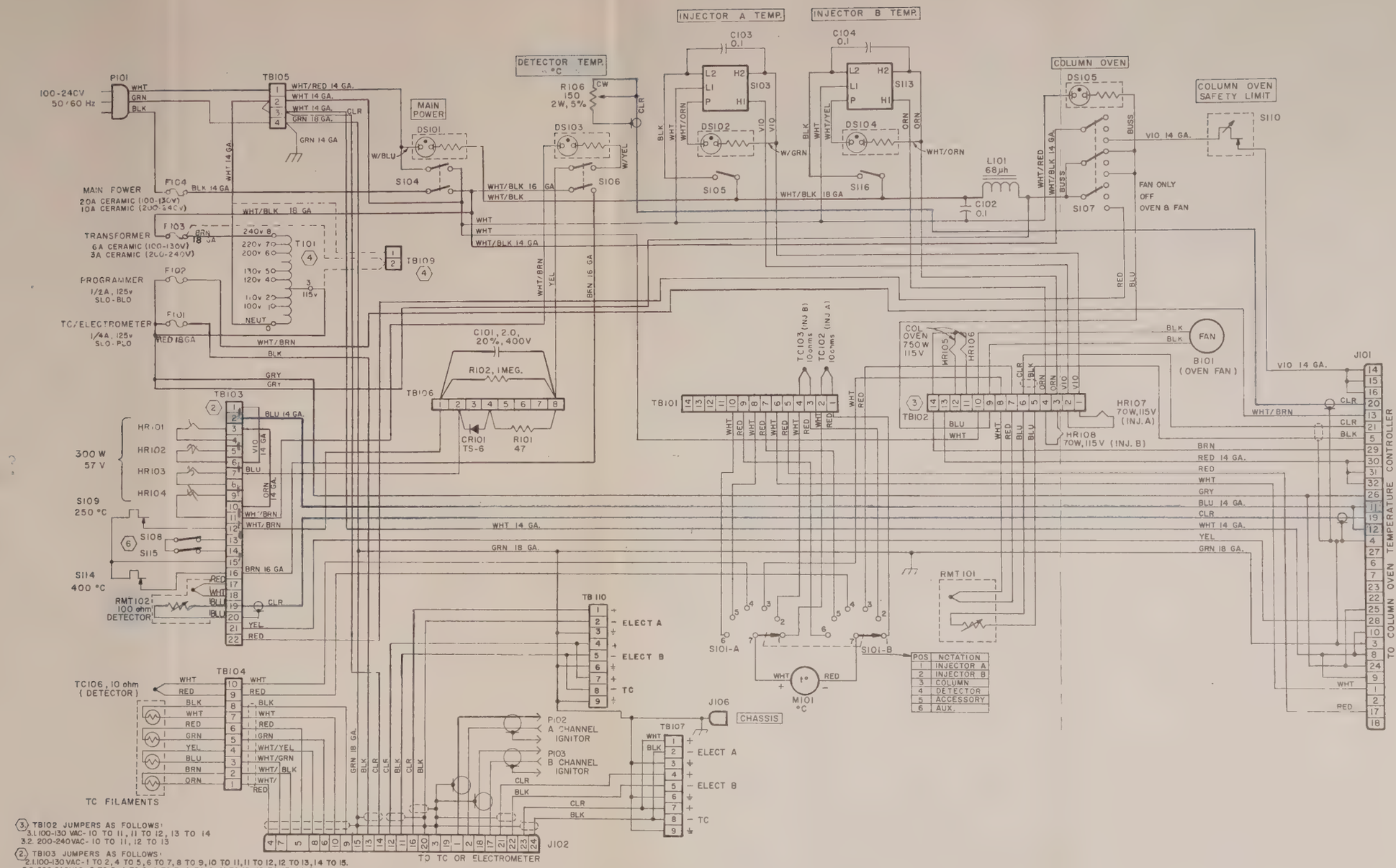
## 5.1 GENERAL

This section of the manual comprises assembly drawings and schematic diagrams.

The parts are identified on the assembly drawings by an item number within a circle or the schematic reference number. The item number or schematic reference number is listed numerically or alphabetically in the parts list. The numbers are followed by a corresponding part number and material description. Use the ten-digit part number and material description when ordering replacement parts. Also, when ordering parts, include the complete Model and Serial numbers of your instrument.

Listed below are the titles and drawing numbers for the assembly and schematic diagrams in this manual.


| TITLE | DWG. NO. | PG. NO. |
|---|---|---|
| 2700 Series Schematic Diagram | 75-000368-00 | 5-2 |
| Front Panel Electrical Components/Hardware Location Drawing | ——— | 5-3 |
| Rear Panel Electrical Components/Hardware Location Drawing | ——— | 5-4 |
| Internal Electrical Components/Hardware Location Drawing | ——— | 5-5 |
| Detector/Column Ovens Electrical Components/ Hardware Location Drawing | ——— | 5-6 |




3. TB102 JUMPERS AS FOLLOWS:
   3.1. 100-130 VAC- 10 TO 11, 11 TO 12, 13 TO 14
   3.2. 200-240 VAC- 10 TO 11, 12 TO 13

2. TB103 JUMPERS AS FOLLOWS:
   2.1. 100-130 VAC- 1 TO 2, 4 TO 5, 6 TO 7, 8 TO 9, 10 TO 11, 11 TO 12, 12 TO 13, 14 TO 15.
   2.2. 200-240 VAC- 2 TO 3, 4 TO 5, 6 TO 7, 8 TO 9, 11 TO 12, 12 TO 13, 14 TO 15.

1. ALL CAPACITORS IN MICROFARADS, ± 20 %, 500 WVDC. ALL RESISTORS IN OHMS, ± 10%, 1/4 WATT, UNLESS OTHERWISE SPECIFIED.

NOTES:

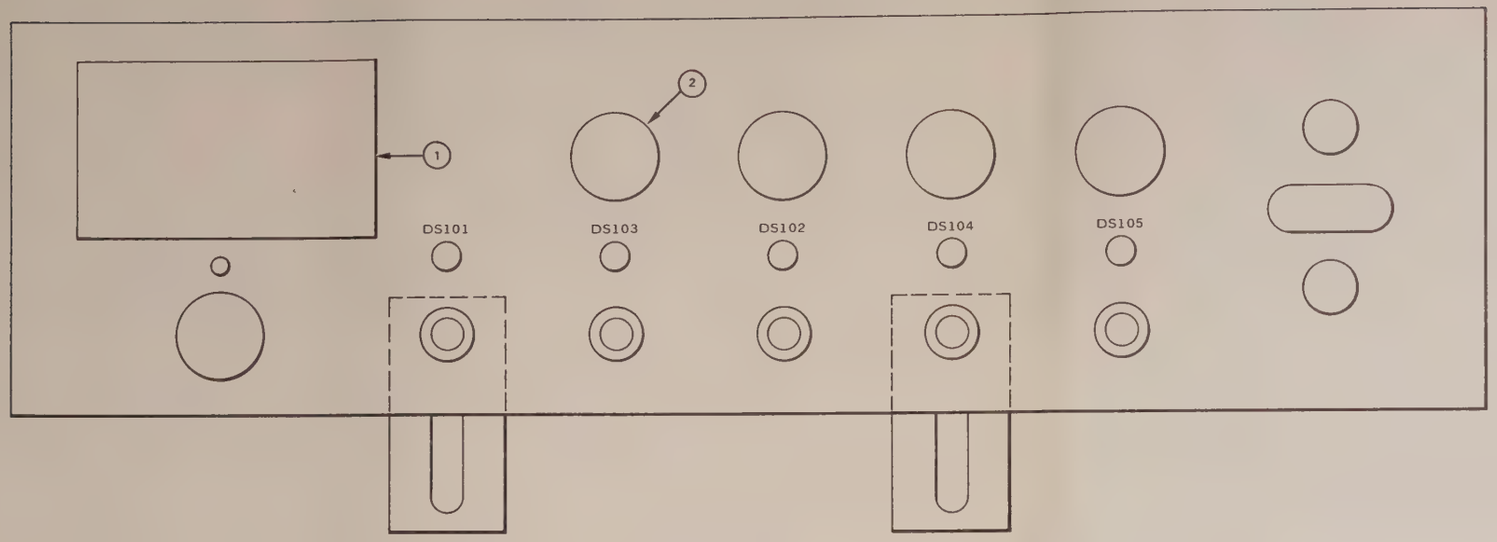
5. ALL WIRES 22 GA. UNLESS OTHERWISE NOTED.

4. T101 USED FOR LINE VOLTAGES OTHER THAN 115V. TB108 USED FOR 115V AND WIRES CONNECTED PER DASHED LINES.

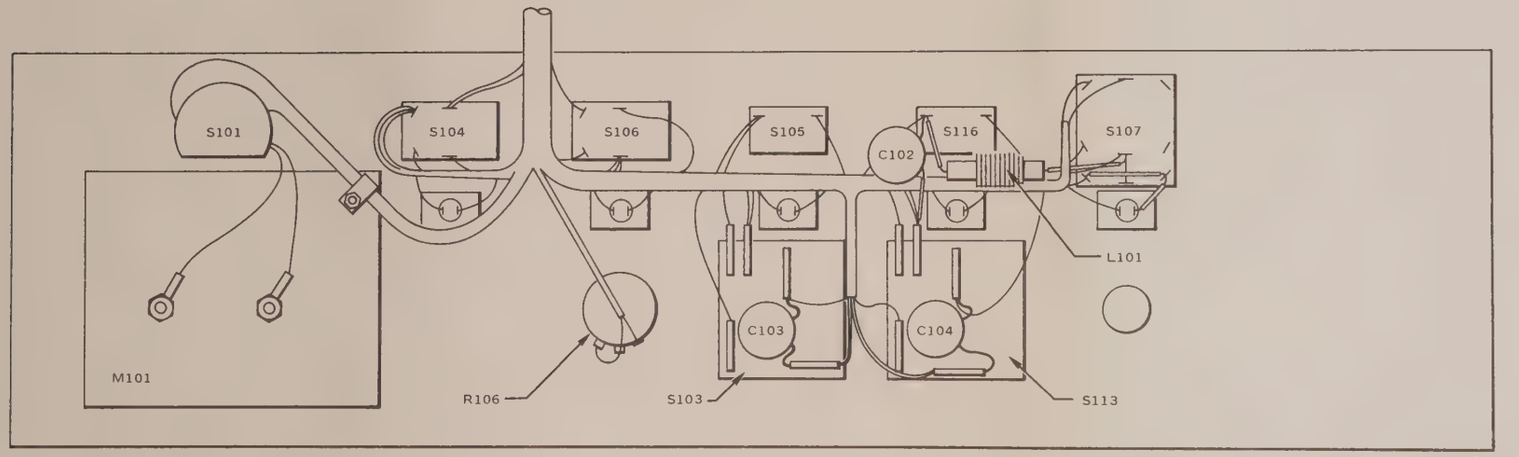
7. FOR ASSY DWG SEE 00-270000-00.

6. S108 AND S115 DELETED AND JUMPER PLACED BETWEEN TB103-13 AND 14 ON 2720 SERIES.

**2700 SERIES SCHEMATIC DIAGRAM**
**75-000368-00 Issue: 2**



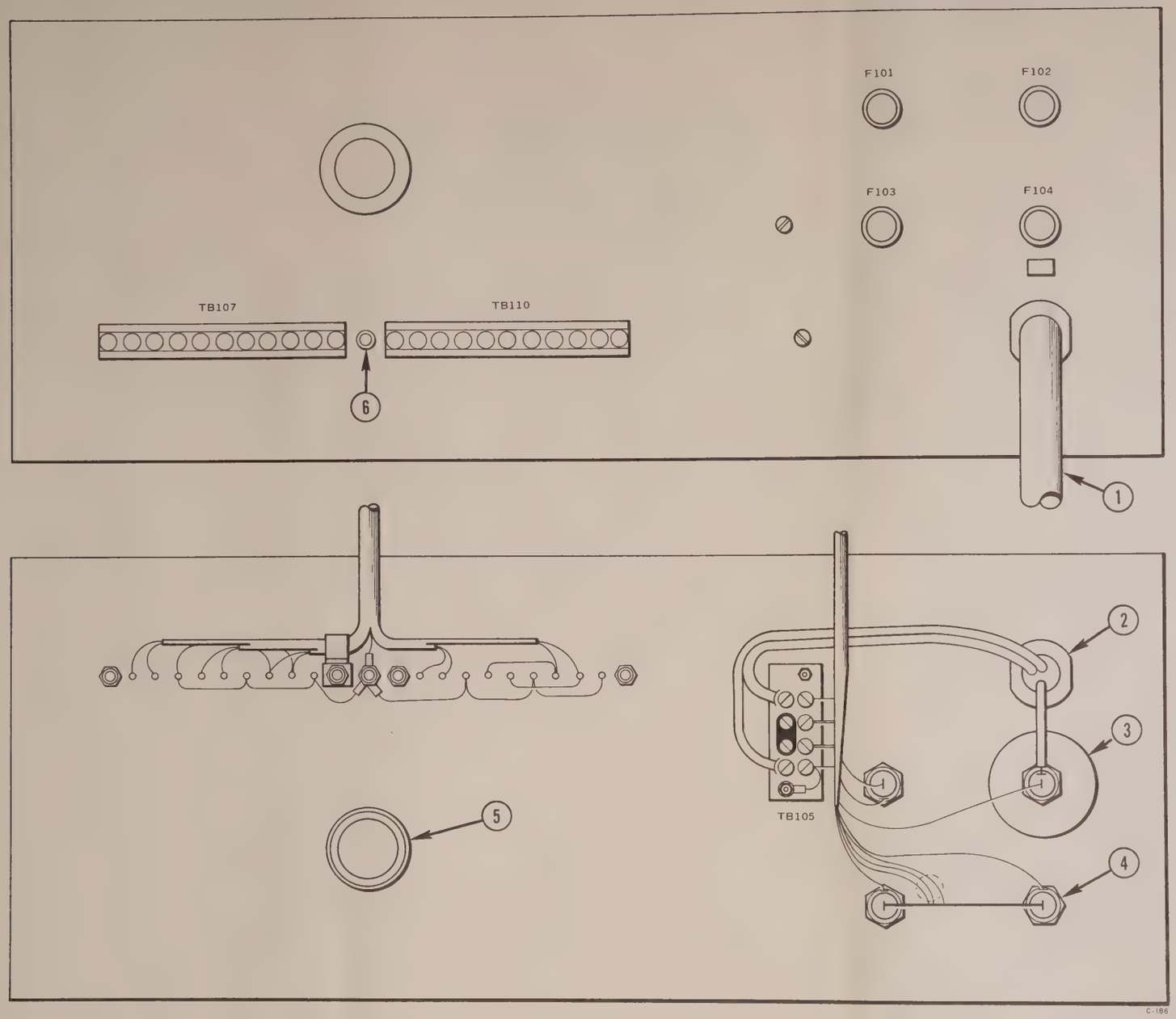

FRONT VIEW




REAR VIEW

| Parts List/ Schematic Reference Number | VA Part No. | Description |
|---|---|---|
| C102 | 05-000003-00 | Capacitor, Disc, .1 μfd, 20%, 5000V |
| C103 | | Same as C102 |
| C104 | | Same as C102 |
| DS101 | 35-000077-00 | Pilot Light, Red, 115 Vac |
| DS102 | | Same as DS101 |
| DS103 | | Same as DS101 |
| DS104 | | Same as DS101 |
| DS105 | | Same as DS101 |
| L101 | 53-000402-00 | Choke, 68 μh, 2A |
| M101 | 29-000069-00 | Pyrometer, 0-400°C |
| R106 | 39-000518-00 | Potentiometer, W.W., 150 Ω , 5%, 2W |
| S101 | 48-000080-00 | Rotary Switch, 2P, 6 Pos. |
| S103 | 48-000048-00 | Regulator, Infinite Input |
| S104 | 48-000102-00 | Toggle Switch, DPST |
| S105 | 48-000086-00 | Toggle Switch, SPDT |
| S106 | | Same as S104 |
| S107 | 48-000103-00 | Toggle Switch, 3PDT |
| S113 | | Same as S103 |
| S116 | | Same as S105 |
| 1 | 35-000020-00 | Half Bezel, 3½" Meter |
| 2 | 25-000017-00 | Knob, Skirted |

**FRONT PANEL ELECTRICAL COMPONENTS/HARDWARE LOCATION DRAWING** Issue: New

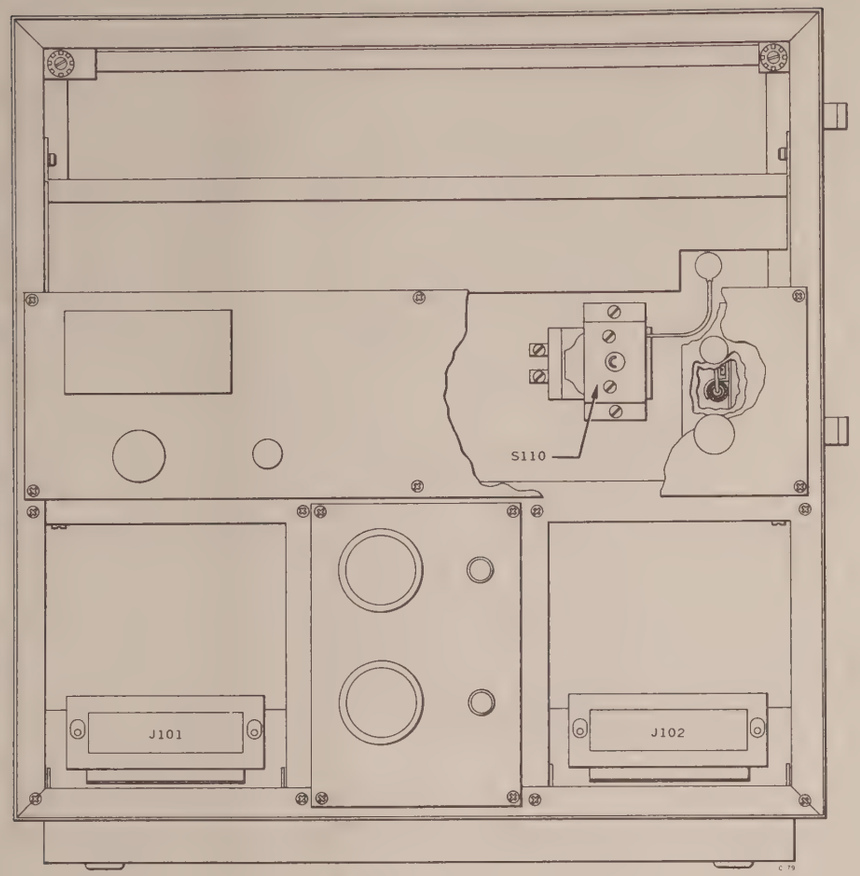



REAR VIEW

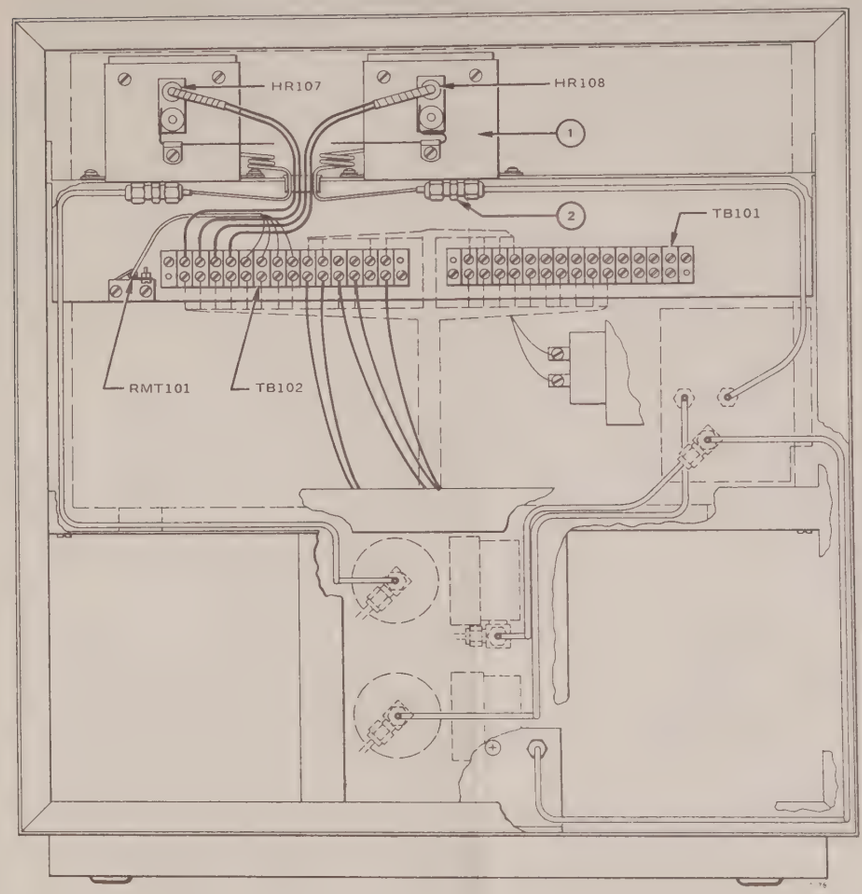
| Parts List/ Schematic Reference Number | VA Part No. | Description |
|---|---|---|
| F101 | 19-000024-00 | Fuse, 1/4A, Slow Blow, 115 Vac |
| F102 | 19-000011-00 | Fuse, 1/2A, Slow Blow, 115 Vac |
| F103 | 19-000046-00 | Fuse, 6A, Ceramic, 250V, 3AB |
| | 19-000045-00 | 3A, Ceramic, 250V, 3AB |
| F104 | 19-000018-00 | Fuse, 20A, Ceramic, 115 Vac |
| | 19-000019-00 | 10A, Ceramic, 230 Vac |
| J461 | 02-000606-00 | Signal Cable (not shown) |
| J462 | | Same as J461 |
| P102 | 01-000680-00 | Ignitor Cable (not shown) |
| P103 | | Same as P102 |
| TB105 | 51-000405-00 | Terminal Block, 4 Positions |
| TB107 | 51-000425-00 | Terminal Block, 9 Positions |
| TB110 | | Same as TB107 |
| 1 | 59-000355-00 | Power Cord, 3 Wire, 12 GA |
| 2 | 15-000865-00 | Strain Relief |
| 3 | 31-000260-00 | Dial Plate |
| 4 | 19-000200-00 | Fuse Holder |
| 5 | 15-000892-00 | Bushing, Snap |

**REAR PANEL ELECTRICAL COMPONENTS/ HARDWARE LOCATION DRAWING** Issue: New

| Parts List/ Schematic Reference Number | VA Part No. | Description |
|---|---|---|
| HR107 | 02-000287-00 | Cartridge Heater Assembly |
| HR108 | | Same as HR107 |
| J101 | 12-000060-00 | Connector, Female, 32 Pins |
| J102 | 12-000057-00 | Connector, Female, 24 Pins |
| RMT101 | 64-000016-00 | Temperature Probe Assembly |
| S110 | 48-000179-00 | Thermostat |
| TB101 | 51-000408-00 | Terminal Block, Barrier, 14 Pos |
| TB102 | | Same as TB101 |
| 1 | 02-001475-00 | Injector Assembly, ¼" |
| 2 | 16-000042-00 | Union, Brass, 1/8-in tubing |



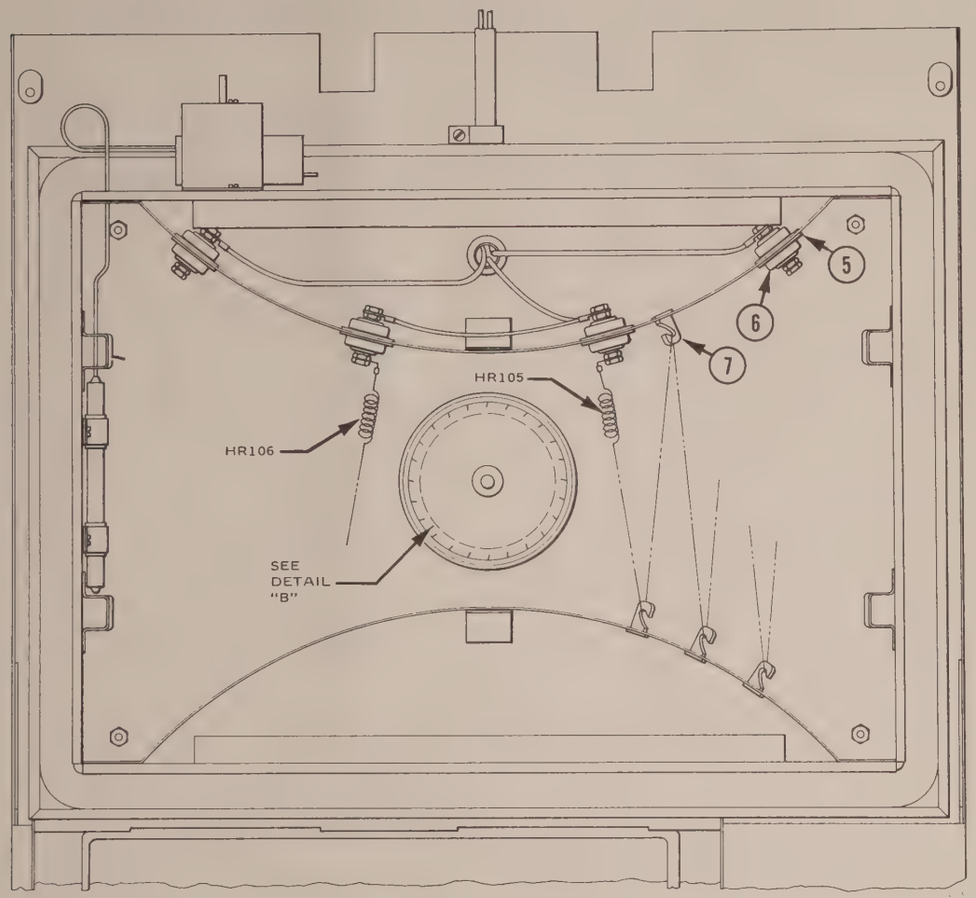

FRONT VIEW



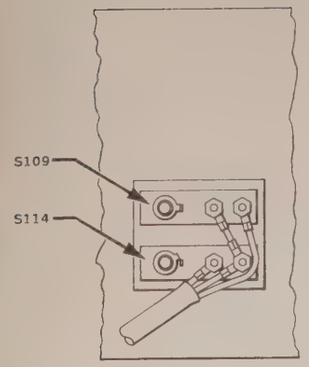

**INTERNAL ELECTRICAL COMPONENTS/HARDWARE LOCATION DRAWING** Issue: New

| Parts List/ Schematic Reference Number | VA Part No. | Description |
|---|---|---|
| B101 | 30-000070-00 | Motor, 115V |
| HR101 | 22-000049-00 | Cartridge Heater, 300W, 57V |
| HR102 | | Same as HR101 |
| HR103 | | Same as HR101 |
| HR104 | | Same as HR101 |
| HR105 | 01-000411-00 | Heater Coil Subassembly, 750W |
| HR106 | | Same as HR105 |
| RMT102 | 64-000009-00 | Temperature Probe |
| S108 | 48-000176-00 | Microswitch |
| S109 | 48-000076-00 | Thermoswitch, 260°C |
| S114 | 48-000104-00 | Thermoswitch, 370°C |
| S115 | | Same as S108 |

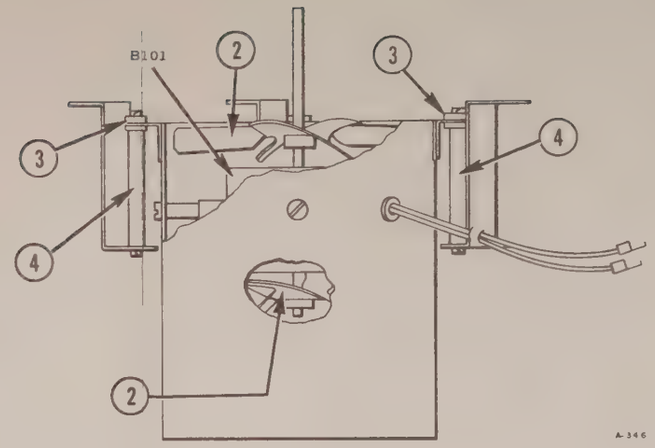
| | | |
|---|---|---|
| TB103 | 51-000410-00 | Terminal Strip, 22 Pos. |
| 1 | 08-000156-00 | Probe and Htr Retainer |
| 2 | 18-000017-00 | Blower Fan |
| 3 | 42-000231-00 | Standoff Rubber |
| 4 | 42-000237-00 | Spacer, Nylon |
| 5 | 15-000722-00 | Mica Washer |
| 6 | 21-000003-00 | Bushing, Feed Thru |
| 7 | 21-000004-00 | Porcelain Insulator |




TOP VIEW

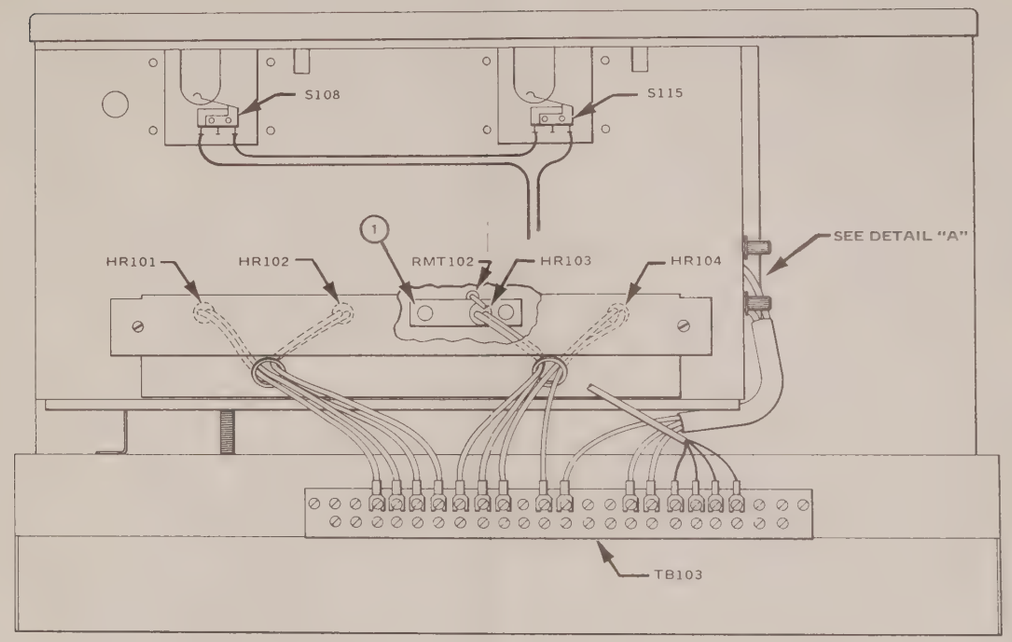



DETAIL "A"




A-346

DETAIL "B"




REAR VIEW

DETECTOR/COLUMN OVENS ELECTRICAL COMPONENTS/HARDWARE LOCATION DRAWING
Issue: 1

# SYRINGE HANDLING TECHNIQUES

In chromatography, your results are often only as good as the reproducibility of your sample. And the reproducibility of your sample quantity depends on many factors, among which are an accurate syringe and the operator.

To help chromatograph operators obtain the greatest reproducibility possible, we have compiled some syringe handling techniques from experienced operators in the field. No suggestion or hint here is "the" answer to every problem, but perhaps you'll find an answer to the problem that's been plaguing you.

**A. Filling the syringe**

1. Be certain the syringe and plunger are clean before filling. Following is one recommended method of cleaning a syringe: Pump cold chromic acid* solution through the syringe with the plunger, rinse both with distilled water, blow the syringe dry and carefully wipe the plunger with lint-free tissue being careful not to touch the plunger shaft with your fingers.

   The chromic acid solution does a good job of destroying organic detergent residues and finger-prints on the plunger.

   The more stubborn oxide stains in the syringe body neck may be removed with aqua regia, but is recommended only as a last resort and should never contact the plunger or needle.

2. Pressurize your sample bottle by using a gas tight syringe filled with inert gas. Repeat as needed to build pressure in the bottle.
   a. This is particularly recommended for syringes with a detachable needle.

3. To assure an accurate measurement, wet the interior surfaces (barrel and plunger) of the syringe with the sample by pumping the plunger before filling. When properly done, this neutralizes the liquid movement by capillary forces.

4. Overfill the syringe in the sample bottle, withdraw from bottle and move plunger to desired calibration line, discharging excess sample. This is your best assurance of a "full" sample.
   a. Discharging excess sample while the needle is still in the sample bottle may cause loss of sample upon withdrawing.

5. Read the syringe graduation from the same angle each time you fill the syringe. Recommended practice is to read the sample at the "top" (flange end) of the calibration line. This provides an accurate visual check, and reduces the problems of "line thickness."
   a. Strive to develop a smooth uniform loading operation to minimize slight involuntary errors.

6. Take a visual check of the syringe for bubbles or foreign matter in the sample.

7. Syringes with a detachable needle require care when filling because of the dead volume in needle. (Refer to point 2a.)

8. Before injecting, wipe needle clean with a lint-free tissue in a quick motion, taking care not to wipe sample out of needle and not to transfer body heat from fingers to needle.

**B. Injecting the sample**

1. Develop a rhythm in your motion that is used each time you inject, i.e. do the same things in the same manner at the same time.

2. Hold the syringe as close to the flange (in the unmarked area) as possible. This will prevent the heat transfer that occurs when you hold the needle or barrel with your fingers.
   a. Another way to prevent heat transfer is to use a Kel-F syringe guide. The guide also makes septum penetration easier.

3. Handle plunger by the button, not the plunger shaft. This reduces the possibility of damage or contamination.

4. Develop a smooth rhythm that allows you to inject the sample as quickly as you can with accuracy.

5. For greatest accuracy, it is recommended that the syringe not be used at maximum capacity.

**C. Some unusual techniques**

1. Some operators have been successful by pulling air into the syringe before filling with sample. This permits a full volume injection with no dead volume in the needle.

2. Another variation of the above technique is to pull air before a wet plunger, adjust the volume, wipe the needle clean, and withdraw the plunger a few microliters. This places the sample between two air blocks.

3. Other operators use "The Difference" technique. Here the syringe is filled in a normal manner, the needle is wiped, the injection is made and needle withdrawn from the instrument. The syringe plunger is immediately withdrawn a few tenths of a microliter and the residue liquid hold-up is read. This hold-up is subtracted from the original reading, giving the net volume of liquid displaced.

4. Still other operators, in the fraction-of-a-microliter range, successfully use "The Needle Boil Off" technique. Here a syringe (i.e. 701N) is wet with sample and the plunger is at zero. The needle then is inserted in the septum for a "time unit" and withdrawn. Some chemists have an excellent mental "time unit" and get good reproducibility.

**D. Summary**

1. Be sure your syringe is clean.
2. Develop a smooth, quick, uniform action.
3. Protect your syringe from damage and contamination.
4. Experiment with these suggestions until you've developed your own successful syringe handling techniques. (a. Then tell us about it, so we can share it with others.)

**Solution: 40 gms chromic anhydride .6cc (1.1 gms) sulfuric acid con. (max.) water to 100 cc.)*

*We are grateful to Hamilton Company, Inc. for the above syringe handling techniques. Varian Aerograph carries a complete line of Hamilton syringes for gas chromatography; see Accessory Catalog.*

# PARKER CPI TUBE FITTINGS

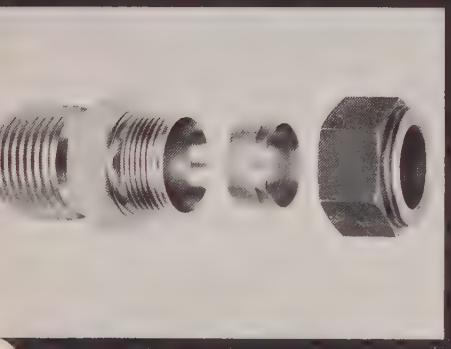
# INSTALLATION PROCEDURE

**Bulletin No. 4230-B10**
**August 1970**

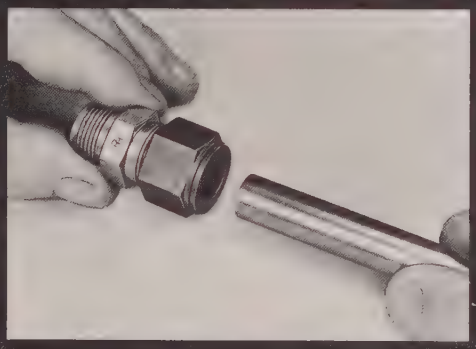
Supersedes Bulletins
4230-B9
4330-B10

**CAUTION** Field surveys indicate that most flareless instrumentation fittings are made up by torque or "feel". This is an absolutely unreliable method of make up because of variables caused by surface finish, tolerances in the manufacture of fittings and tubing, cleanliness of the fitting at installation, number of threads engaged between fitting nut and body, wall thickness of the tube and lubricity of the lubricant used.

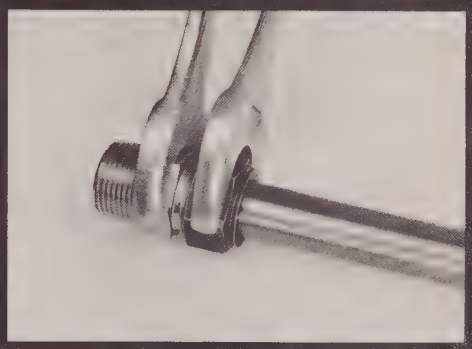
By design, *all* such instrumentation fittings are to be made up by counting rotation of the nut. This assures the required movement of the ferrule(s) in relation to the tube. Please follow the directions below.



It is not necessary to disassemble the fitting. If the fitting is disassembled, note that the small tapered end of the ferrule goes into the fitting body.



Insert tube as illustrated until the tube bottoms against the shoulder in the fitting body. Care should be taken to insure the tube is in proper alignment with fitting body.



Tighten nut finger tight. Then tighten with wrench an additional 1¼ turns. It is recommended that the nut be marked to facilitate counting the 1¼ turns.*
*Except for 1/16" & 1/8" O.D. tube

**TUBE SELECTION.**
For best results follow the below recommendations. Care in handling should be used to avoid scratching or abrading the surface of the tube.

**STAINLESS STEEL.**
Use type 304 or 316 stainless steel seamless, fully annealed and pickled (e.g. ASTM A269). Maximum hardness should not exceed Rockwell B90.

**STEEL.**
Use seamless fully annealed steel (e.g. SAE 1010). Maximum hardness not to exceed Rockwell B-65.

**COPPER & ALUMINUM.**
These materials can be used up to the half hard condition although fully annealed material will give the best results.

**SEATING THE TUBE.**
It is important to insert the tube into the fitting until it bottoms out against the built-in tube stop. This is necessary to stop movement of the tube while establishing the slight "bite" of the ferrule through any imperfections that may exist on the outside tube surface.

Normal make up recommendation is 1¼ turns (for 1/16" & 1/8" tube use ¾ turn) from finger tight. For high pressure tighten an additional ¼ turn.

**Normal Pressure 1¼ Turns** **High Pressure 1½ Turns**

**REMAKE INSTRUCTIONS.**
Mark the fitting with a line (A-A') to show the relationship of the nut and fitting body before disassembly. Remake by retightening until marks line up and then tighten a slight additional amount. (Line B aligned with line A) The highest number of remakes will result, if these instructions are followed.

**ALTERNATE INSTRUCTIONS: SMALL SIZES**

In some cases, it is desirable to alter the standard make-up procedure for Parker CPI tube fittings. Recommended variations are shown below

NOTE: The make-up for size 1 is a permanent instruction and is not optional.

| TUBE SIZE | MAKE-UP PROCEDURE |
|---|---|
| 1/16 & 1/8 | Three-fourths (¾) turn from finger-tight. |
| 3/16 & ¼ | Standard 1¼ turn from finger-tight is normal recommendation; however, if it is desirable to reduce deformation of tube I.D. use three-fourths (¾) turn from finger-tight. Example: Thermocouple fittings. |
| ⅜ up | Standard 1¼ turn from finger-tight is normal recommendation; however, if it is desirable to reduce deformation of tube I.D. use 1 turn from finger-tight. Example: Thermocouple fittings. |



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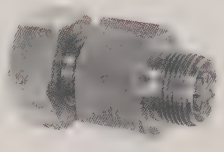
The Parker CPI tube fitting has been specifically designed for use in the chemical process industries, but will also find use in other fields where a high quality tube fitting is required. The Parker CPI fitting is available in brass, type 316 stainless steel, steel and aluminum. They can be furnished in almost any machineable material.

Parker CPI tube fittings' highly refined component design coupled with the Parker Hannifin "Black Nut" for stainless steel provides exceptionally low-torque on assembly. The "Black Nut" is pre-lubricated with a bonded dry-film lubricant, making it unnecessary to use additional thread lubrication. Torque is reduced as much as 50% in the case of stainless steel.



## INSTANCES WHERE PRESETTING CPI FITTING IS RECOMMENDED

Presetting Parker CPI ferrules is not essential; however, there are some instances where presetting is recommended. Two of these conditions are:

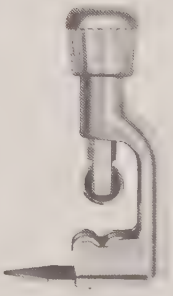
1. Thin wall tubing or situations requiring maximum bore for through flow applications. In this case, the presetting tool with expanding mandrel should be used.

2. On installations requiring a large number of disconnects and reassembly. Also, on connections to valves since presetting extends the life of both fitting or valve bodies. Presetting in this instance can be done with a presetting tool without use of the expanding mandrel.

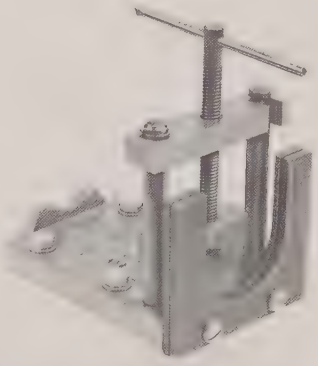
### HOW TO PRESET

Select proper size presetting tool to correspond to size of fitting, slide nut and then ferrule onto tube. (Note that the small tapered end of the ferrule goes into the tool body). Set tube into preset tool making sure that the tube end is against the shoulder at the bottom of the tool. If the expanding mandrel is used (sizes—8 thru 16), wrench the bottom hex of the tool to expand the mandrel to snugly fit the I.D. of the tubing. Next, bring nut and ferrule down and screw on finger tight. Tighten with wrench ¾ of the turn during which time the tube is held firmly against the tool shoulder.

Remove tube assembly from presetting tool and assemble to fitting or valve body, in installation, by tightening the nut down finger tight then wrench tight the nut until a rapid rise in torque is noted. From this point turn the nut approximately ½ turn more.

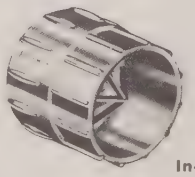


Hand Tube Cutter



Saw-Skware-Hacksaw Guide

Hand Tube Benders



In-Ex Tube Deburring Tool

**TUBE FITTINGS DIVISION**

PARKER HANNIFIN

17325 Euclid Avenue, Cleveland, Ohio 44112

varian aerograph

2700 mitchell drive/walnut creek/california 94598/415-939-2400
specialists in chromatography and data handling systems

# LINEAR TEMPERATURE PROGRAMMER

# INSTRUCTION MANUAL

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LINEAR TEMPERATURE PROGRAMMER

# SECTION 1
# INTRODUCTION

## 1.1 GENERAL

The Varian Aerograph™ Linear Temperature Programmer (LTP) provides precise linear temperature programming or isothermal operation of the column oven in a gas chromatograph. The LTP is primarily designed for use in the 2700 and 2800 Series instruments. However, an outboard version of the LTP (Model 330) is available for use with other Varian Aerograph instruments such as the Models 550, 600, 700, 701, 90-P, 90-P3, and 90-P5. Instructions for interconnecting the Model 330 to the above listed instruments are included in the appendix as are instructions for interconnecting the Model 330 to equipment other than Varian Aerograph instruments. Operation of the LTP is the same in all cases.

Refer to Table 1-1 for LTP specifications.

TABLE 1-1
SPECIFICATIONS

**Modes:**
Isothermal; program to preset limit and hold or cool to ambient.

**Temperature Range:**
Ambient to 400°C.

**Stability:**
± 0.5°C by full proportional control.

**Linear Programming Rates:**
0.5, 1, 2, 4, 6, 8, 10, 12, 15, and 20°C per minute; other rates between 0.5 and 20°C with auxiliary plugs.

**Temperature Control**
Direct dialing and readout of initial and upper limit temperatures.

# SECTION 2
# OPERATION

## 2.1 GENERAL

The Linear Temperature Programmer controls the column-oven temperature either isothermally or by programming at a selected linear rate. Refer to Figure 2-1 and accompanying description of controls found on module.

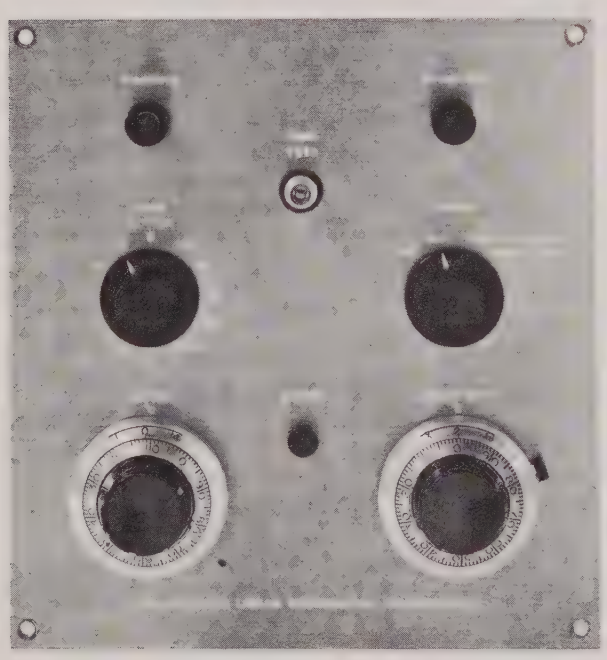


FIGURE 2-1 LINEAR TEMPERATURE PROGRAMMER MODULE

MODE Switch
Selects mode of operation for controlling the column-oven temperature.
OFF position: Turns off power to programmer.
ISO position: Column oven is held isothermally at temperature setting of INITIAL °C control.
HOLD position: Column oven is held isothermally at temperature setting of UPPER LIMIT control at end of program.
COOL position: Power to column-oven heater is turned off at end of program and oven allowed to cool to ambient.

INITIAL °C Control
Sets initial or starting temperature of a program. The control is also used for setting isothermal temperature when using ISO mode of operation. During temperature programming, control turns at rate selected by RATE °C/MIN switch. Control reads directly in °C.

POWER Light
Lights when power is applied to programmer. Power to instrument must be on and the PROGRAM MODE switch set to ISO, HOLD, or COOL positions before light turns on.

UPPER LIMIT °C Control
Sets upper temperature limit of a program. Control reads directly in °C.

PROGRAM Light
Light indicates only when program is in progress.

RATE °C/MIN Switch
Selects °C rate of temperature increase per minute during a program.
AUX position: Switches AUX RATE jack into circuit.

AUX RATE Jack
Permits selection of programming rates other than those selected by the RATE °C/MIN switch. RATE °C/MIN switch must be in AUX position and an optional rate plug inserted into AUX RATE jack. Programming rates selected must range between .5 and 20°C/min. Refer to paragraph 2.2.4 for rate plug calculations.

HEATING Light
Indicates when power is supplied to column-oven heater.

## 2.2 OPERATION

The LTP offers both isothermal and linear temperature programming modes of operation. In the programming mode, the column oven can be held isothermally or allowed to cool to ambient at the end of the program.

The following paragraphs outline procedures for operating the LTP. Note that certain controls or switches on the 2700 or 2800 Series Basic Instrument, e.g., MAIN POWER switch, COLUMN SAFETY LIMIT °C control, COLUMN OVEN switch, must be in certain positions before the LTP can be operated. Refer to the 2700 or 2800 Series Basic Instrument Manual for proper settings of these controls. If the LTP is operated as an outboard programmer, refer to appropriate manual of instrument being used with LTP for settings of controls affecting LTP operation.

### 2.2.1 Isothermal Operation

To operate column oven isothermally, proceed as follows:

1) Set MODE selector to ISO position. This turns on power to programmer circuits and places programmer into the appropriate mode of operation. The POWER light on the programmer should turn on, indicating power is on.

2) Set INITIAL °C control to desired isothermal temperature. Control reads directly in °C. Column oven will now heat to selected isothermal temperature. The HEATING light will turn on brightly until the isothermal temperature is reached, then dim and brighten at varying rates as the oven temperature is maintained.

3) To turn off programmer, set MODE switch to OFF position.

### 2.2.2 Linear Temperature Programming Operation

The column-oven temperature can be programmed at a linear rate from a preestablished isothermal temperature to a preset upper temperature, then automatically cooled to ambient by using the COOL mode or held isothermally at the upper temperature by using the HOLD mode. Proceed as follows:

1) Set MODE selector to ISO position. This turns on power to programmer circuits and places programmer into the appropriate mode of operation. The POWER light on the programmer should light, indicating power is on.

2) Set INITIAL °C control to desired isothermal temperature. Control reads directly in °C. Column oven will now heat to selected isothermal temperature. The HEATING light will turn on brightly until the isothermal temperature is reached, then dim and brighten at varying rates as the oven temperature is maintained.

3) Set UPPER LIMIT °C control to desired upper temperature limit of program. Control is calibrated in °C.

4) Set RATE °C/MIN control to desired programming rate. If you require a programming rate other than that indicated, set RATE °C/MIN control to AUX position and insert the appropriate optional rate plug in the AUX RATE jack. Refer to paragraph 2.2.4 for discussion on rate plug.

5) Set MODE switch to either HOLD or COOL position to start program. Leave MODE switch in HOLD position if you desire oven to remain in isothermal condition at end of program or switch to COOL position if you desire oven to cool to ambient at end of program. PROGRAM pilot light indicates while program is in progress. Note that the INITIAL °C control turns at a rate which corresponds to the programming rate selected. The INITIAL °C control will continue to run until its dial reading matches the dial reading of the UPPER LIMIT °C control. At that time, the column-oven temperature will be maintained in an isothermal condition (HOLD mode) or allowed to cool to ambient (COOL mode) by power being removed from the column-oven heaters.

### 2.2.3 Program Reset and Successive Program Setup

Proceed as follows:

1) Set MODE switch to ISO position.

2) Reset INITIAL °C control to desired isothermal starting temperature.

NOTE

**UPPER LIMIT °C control does not require resetting unless new upper temperature is required.**

3) To facilitate column oven cool-down, open column-oven door. Allow oven temperature to drop about 15 to 20°C below isothermal starting temperature. Monitor temperature on pyrometer located on main control panel of basic instrument.

4) Close column-oven door.

5) Allow column-oven temperature to equilibrate at temperature selected by INITIAL °C control. Monitor temperature on pyrometer (located on main control panel of basic instrument).

6) This completes reset and setup of programmer. To start next program, set MODE switch to HOLD or COOL position.

**2.2.4 Calculation of Auxiliary Rate Plug Resistance**
Programming rates other than those provided by the LTP RATE °C/MIN switch are possible with the use of optional auxiliary rate plugs. The rates selected must lie between .5 and 20°C/Min. Each auxiliary rate plug requires a precision resistor, the value of which determines the programming rate in °C per minute. Blank (without resistor installed) rate plugs are available from Varian Aerograph (Part No. 02-000503-00). To calculate a resistance value, use the following formula.

Formula: $R = A/V - R_1$

A = constant $500 \times 10^3$ ohms,

V = desired rate in °C/Min,

R = resistance value in ohms, and

$R_1$ = 10,000 ohms.

**Example:**

Where: desired rate V = 5°/minute

$$R = \frac{500 \times 10^3}{5} - 10 \times 10^3 \text{ ohms}$$

$R = (100 - 10) \times 10^3$ ohms

$R = 90 \times 10^3$ or

R = 90 Kohms

NOTE

**The resistor recommended is a type CEC (T-O) 1% metal film manufactured by International Resistance Company (or equivalent).**

# SECTION 3
# SERVICING

## 3.1 GENERAL

The following paragraphs discuss the calibration procedures necessary to ensure proper operation of your instrument.

Some of the calibration procedures require the use of an extension cable. You should purchase this extension cable from Varian Aerograph. The Part No. is 02-001090-00.

The following paragraphs outline procedures for calibrating the INITIAL °C, UPPER LIMIT °C, and RATE °C/MIN controls.

NOTE

**Before making any calibration adjustments on the programmer, check setting of controls against external pyrometer of known accuracy. This will determine if pyrometer on main control panel (located on basic instrument) or control being checked is out of calibration.**

### 3.1.1 INITIAL °C Control Calibration

If settings of the front panel INITIAL °C control do not agree with pyrometer on main control panel, the Isothermal Set control may need recalibration. Recalibrate control as follows:

NOTE

**An extension cable (Part No. 02-001090-00) is necessary to carry out the following calibration procedures.**

1) Turn off power to instrument via MAIN POWER switch located on main control panel of basic instrument.

2) Remove four screws securing Linear Programmer to cabinet. Remove programmer from cabinet.

3) Connect programmer to cabinet with extension cable (Part No. 02-001090-00).

4) Set INITIAL °C control to 200°C. Control reads directly in °C.

5) Set COLUMN SAFETY LIMIT °C control on main control panel to 250°C.

6) Set COLUMN OVEN switch on main control panel to OVEN & FAN position.

7) Turn on power to instrument via MAIN POWER switch located on main control panel.

8) Set MODE selector to ISO position.

9) Monitor column-oven temperature on pyrometer located on main control panel. If column oven does not equilibrate at 200°C, adjust Isothermal Set control until column-oven temperature equilibrates at 200°C. Locate Isothermal Set control from Figure 3-1. Clockwise rotation of control increases temperature; counterclockwise rotation of control decreases temperature. If the INITIAL °C control does not calibrate properly, consult your nearest Varian Aerograph representative for servicing information.

10) Turn off power to instrument and reinstall module in cabinet.

### 3.1.2 UPPER LIMIT °C Control Calibration Procedures

If the settings of the front panel UPPER LIMIT °C control do not agree with INITIAL °C control settings, the Upper Limit Adjust control may need recalibration. Recalibrate control as follows:

1) Make sure INITIAL °C control functions properly as outlined in paragraph 3.1.1.

2) Turn off power to instrument via MAIN POWER switch located on main control panel of basic instrument.

3) Remove four screws securing module to cabinet. Remove programmer from cabinet.

4) Connect programmer to cabinet with extension cable (Part No. 02-001090-00).

5) Set INITIAL °C control to 200°C. Control reads directly in °C.

6) Set RATE °C/MIN switch to AUX position.

7) Set UPPER LIMIT °C control to 250°C. Control reads directly in °C.

8) Set COLUMN SAFETY LIMIT °C control on main control panel to 250°C.

9) Set COLUMN OVEN switch on main control panel to OVEN & FAN position.

10) Turn on power to instrument via MAIN POWER switch located on main control panel.

11) Set MODE selector to HOLD position.

12) Slowly adjust UPPER LIMIT °C control to a lower setting of 200°C. PROGRAM lamp should turn off at 200°C. If lamp turns off before UPPER LIMIT °C control reaches 200°C or does not turn off at 200°C, adjust Upper Limit Adjust control until PROGRAM lamp turns off at 200°C. Clockwise rotation of Upper Limit Adjust control increases temperature; counterclockwise rotation of control lowers temperature. Refer to Figure 3-1 for location of Upper Limit Adjust control. If the UPPER LIMIT °C control does not calibrate properly, consult nearest Varian Aerograph representative for service information.

13) Turn off main power to instrument and reinstall module in cabinet.

**3.1.3 RATE °C/MIN Switch Calibration Procedure**

If the settings of the front panel RATE °C/MIN switch do not agree with the rates as called out on the panel around the switch, the Rate Adjust control may need recalibration. Recalibrate control as follows:

1) Turn off power to instrument via MAIN POWER switch located on main control panel.

2) Remove four screws securing Linear Programmer to cabinet. Remove programmer from cabinet.

3) Connect programmer to cabinet with extension cable (Part No. 02-001090-00).

4) Set INITIAL °C control to 100°C. Control reads directly in °C.

5) Set RATE °C/MIN to 20°C/MIN position.

6) Set UPPER LIMIT °C control to 160°C. Control reads directly in °C.

7) Set COLUMN SAFETY LIMIT °C control on main control panel to 200°C.

8) Turn on power to instrument via MAIN POWER switch located on main control panel.

9) Set MODE selector to ISO position. Allow oven temperature to equilibrate at setting of INITIAL °C control.

10) After temperature has equilibrated at 100°C, set MODE selector to HOLD position. PROGRAM lamp should light indicating program is in progress. Immediately start timing program. PROGRAM lamp should turn off in three minutes ± 3 seconds. If the programming rate does not fall within the limits mentioned above, adjust for proper program rate (20°C/MIN). Clockwise rotation of control increases programming rate; counterclockwise rotation decreases programming rate. One full turn of Rate Adjust control equals approximately 5 seconds. Refer to Figure 3-1 for location of Rate Adjust control. If the RATE °C/MIN control does not calibrate properly, consult nearest Varian Aerograph representative for servicing information.

11) Turn off main power to instrument and reinstall module in cabinet.

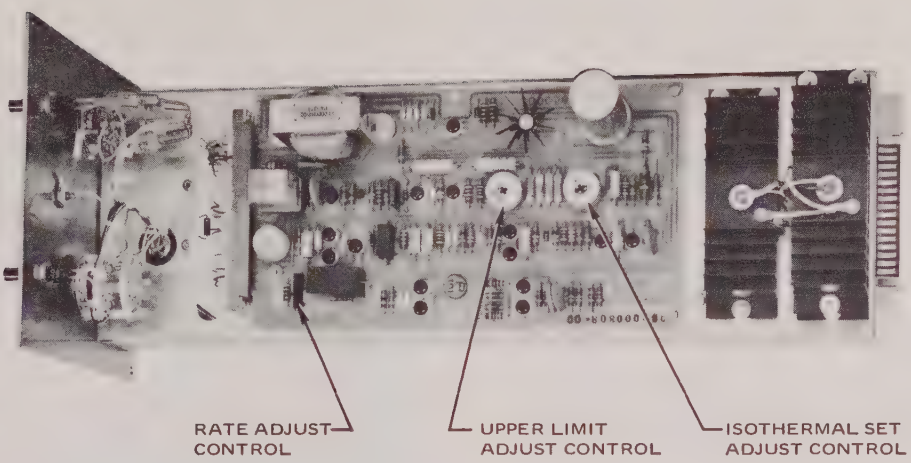



FIGURE 3-1 TOP VIEW OF LINEAR TEMPERATURE PROGRAMMER

## APPENDIX A

The Model 330 is an outboard LTP. As shown in Figure 4-1, the Model 330 is mounted in its own cabinet.

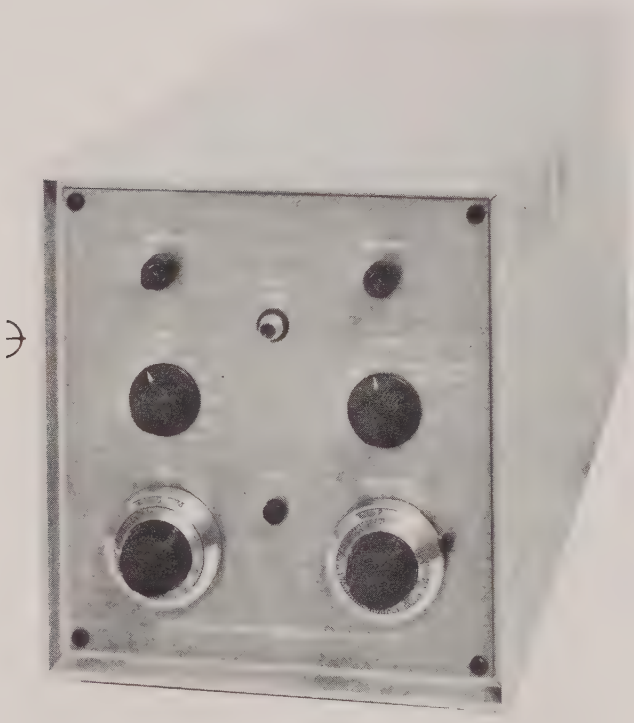


FIGURE 4-1 VARIAN AEROGRAPH MODEL 330

The Model 330 requires up to a 15 amp, 115 volt ac line for operation. A maximum of 1500 watts can be controlled by the Model 330. For optimum performance, the Model 330 should be operated from as steady an ac line voltage as possible. The three-conductor polarized line cord with straight blade plug furnished with the instrument should be plugged into a matching straight-blade receptacle with the correct polarity. The outlet should be wired as shown in Figure 4-2. The polarity may be checked with an ac voltmeter. Approximately 115 Vac will be measured between the hot and ground connections and 0 Vac will be measured between the neutral and ground connections.

NOTE

**If a two-conductor nonpolarized adapter is used, a separate grounding wire must be connected from the Model 330 to a good earth ground.**

The Model 330 can be connected to Varian Aerograph instruments as well as those of other companies. On recent Varian Aerograph instruments, e.g., 600, 700, 701, 90-P3, 90-P5, etc., a Twist-Lock receptacle is provided with the instrument. A quick connection can easily be made between the Model 330 and instrument by just plugging the OUTPUT Twist-Lock connector into receptacle on instrument. On older version Varian Aerograph

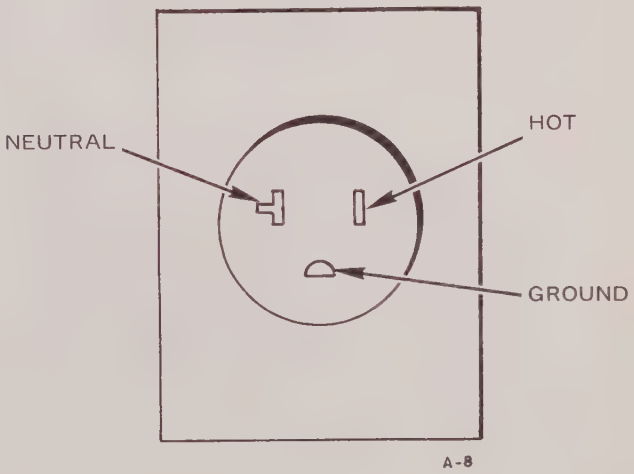



FIGURE 4-2 OUTPUT POLARITY OF STRAIGHT—BLADE RECEPTACLE

instruments as well as instruments of other companies, follow the steps as outlined below:

NOTE

**A conversion kit containing instructions and the necessary hardware is available for Varian Aerograph Models 550, 600, 700, 701, 90-P, 90-P2, 90-P3, and 90-P5 for use with the Model 330.**

1) Identify the three-conductor cable terminated with a female Twist-Lock connector (HEATER POWER OUTPUT) on the rear panel of the Model 330. Refer to Figure 4-3 for identification of connector and output connections.

2) Terminate the connector of the three-conductor cable as follows:

   a) Wire load and neutral leads to heater element in external oven.
   b) Wire ground lead to instrument ground (chassis).

3) Install probe in oven to be controlled (leads from probe connect to Model 330). A 1/4-inch hole must be drilled in the side of the oven in a position permitting the probe, when installed in hole, to be located in area requiring greatest temperature control.

— CAUTION —

**The probe and its housing are fragile. Use caution when installing probe and when working in area of probe to prevent its damage.**

On Models 90-P, P2, P3, P5, 700, and 701, the probe can be inserted into the oven through a 1/4-inch hole which can be drilled next to the air coolant tube. On Models 550 and 600, the probe can be inserted into the oven through the hold available after removing the dial thermometer (dial thermometer located on front panel of instrument).

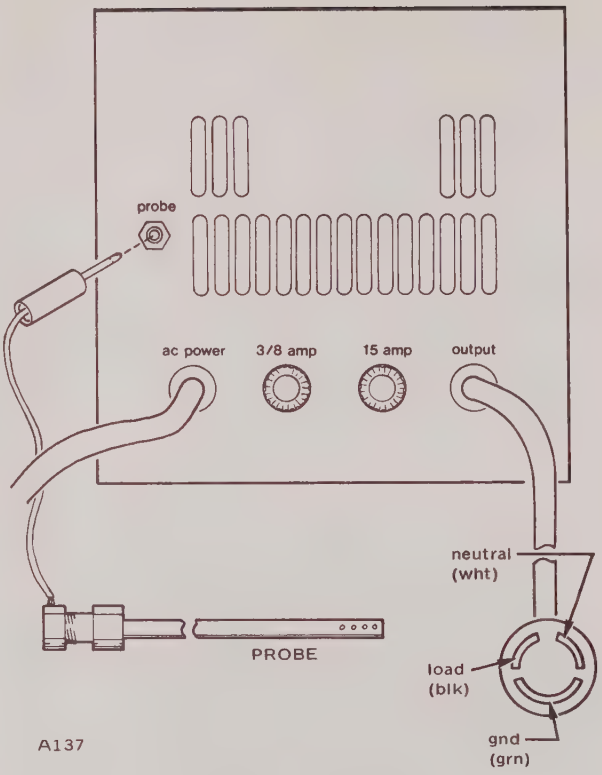


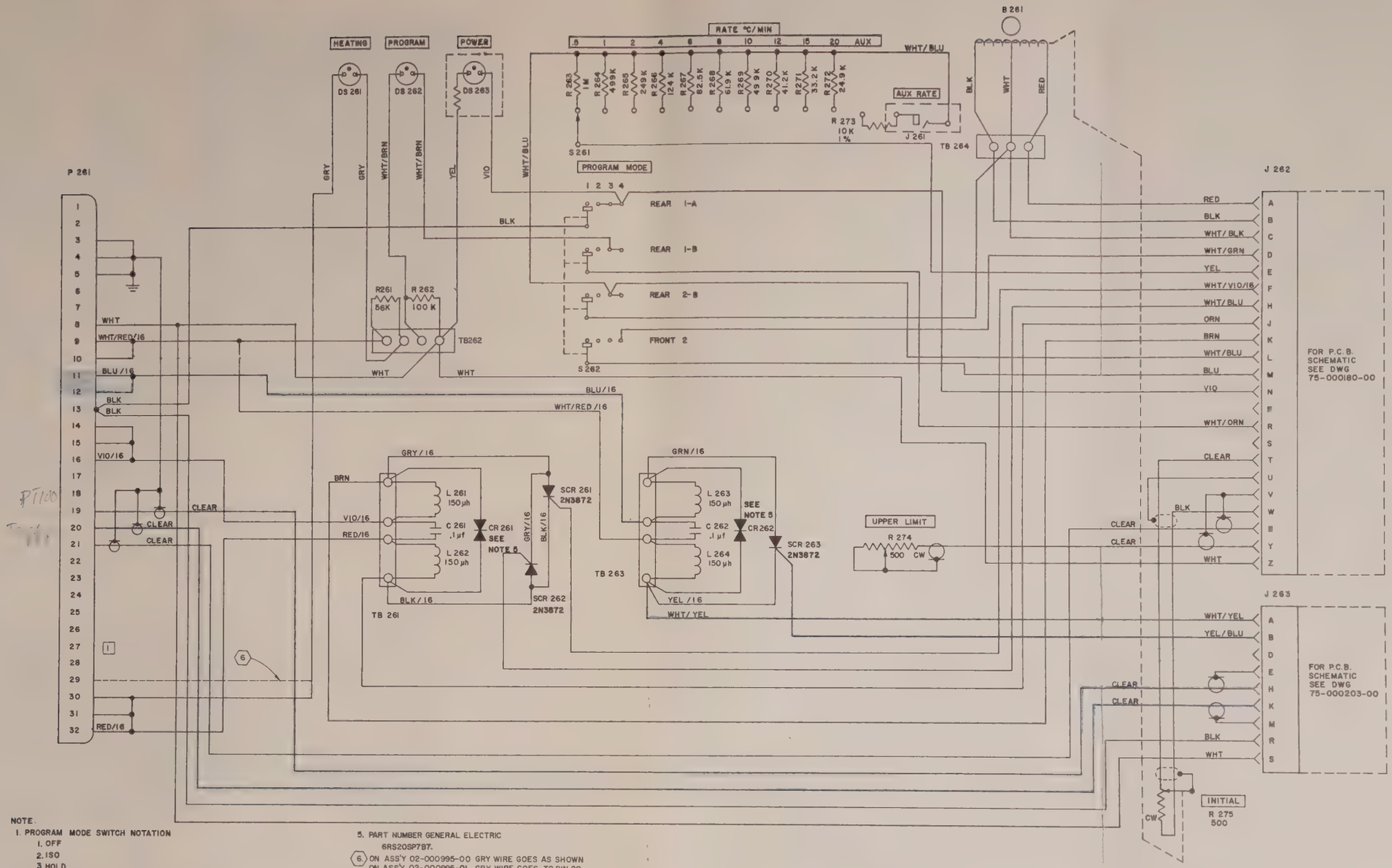

FIGURE 4-3 REAR PANEL

# SECTION 5
# ILLUSTRATED PARTS LISTS/SCHEMATICS

## 5.1 GENERAL

This section of the manual contains the illustrated parts lists and schematic diagrams for the LTP. Listed below in the order they appear are the titles and drawing numbers (where applicable) of all illustrated parts lists and schematic diagrams.


| TITLE | DWG. NO. | PAGE NO. |
|---|---|---|
| Linear Temperature Programmer Schematic Diagram | 75-000224-00 | 5-2 |
| Chassis Electrical Components/Hardware Location Drawing | ——— | 5-3 |
| Temperature Programmer PC Board Schematic Diagram | 75-000180-00 | 5-4 |
| Temperature Programmer PC Board Drawing | 02-000808-00 | 5-5 |
| Isothermal Control PC Board Schematic Diagram | 75-000203-00 | 5-6 |
| Isothermal Control PC Board Drawing | 02-000800-00 | 5-6 |
| Model 330 Interconnection Wiring Diagram | 75-000293-00 | 5-7 |



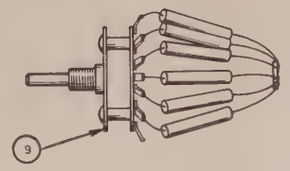

NOTE.

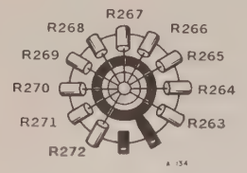
1. PROGRAM MODE SWITCH NOTATION
   1. OFF
   2. ISO
   3. HOLD
   4. COOL
2. ALL WIRES 22 GA EXCEPT WHERE NOTED
3. RESISTANCES IN OHMS
   a. RESISTORS R 261 & R 262 1/2 WATT, 10 % CARBON
   b RESISTORS R 263 THRU R 273 1/2 WATT, 1%, METAL FILM.
   c. RESISTORS R 274 & R 275 WIRE WOUND, 10 TURN
4. FOR ASS'Y DWG SEE 02-000995-00
   FOR ASS'Y DWG SEE 02-000995-01
5. PART NUMBER GENERAL ELECTRIC 6RS20SP7B7.

(6) ON ASS'Y 02-000995-00 GRY WIRE GOES AS SHOWN
ON ASS'Y 02-000995-01 GRY WIRE GOES TO PIN 29

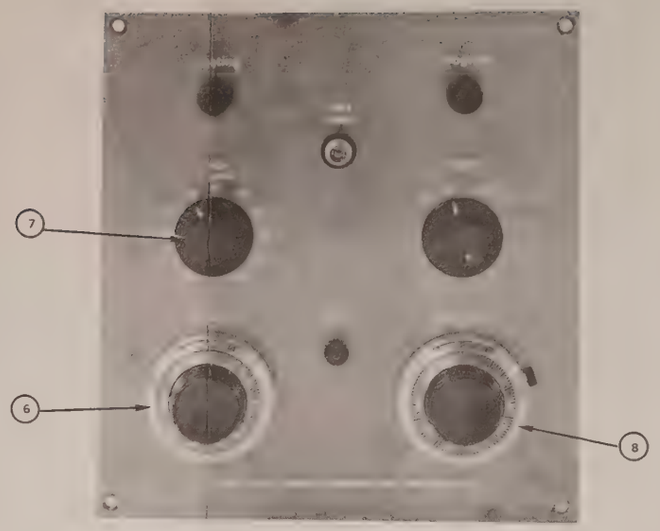
**LINEAR TEMPERATURE PROGRAMMER SCHEMATIC**
**DIAGRAM: 75-000224-00 Issue: 4**

| Parts List/ Schematic Reference Number | VA Part No. | Description |
|---|---|---|
| B261 | 30-000024-00 | Pulse Stepping Motor |
| C261 | 05-000003-00 | Capacitor, Disc Ceramic, .1 µfd, 500V |
| C262 | | Same as C261 |
| CR261 | 14-000051-00 | Diode, Thyrector, 200V |
| CR262 | | Same as CR261 |
| DS261 | 56-000519-00 | Neon Lamp-Cartridge, Red, NE-2E |
| DS262 | | Same as DS261 |
| DS263 | 35-000077-00 | Pilot Light, Red, Neon |
| J261 | 12-000114-00 | Phone Jack |
| J262 | 12-000139-00 | Connector, PC Board, 22 Pins |
| J263 | 12-000140-00 | Connector, PC Board, 15 Pin |
| L261 | 01-000336-00 | Choke, Toroidal, Assembly |
| L262 | | Same as L261 |
| L263 | | Same as L261 |
| L264 | | Same as L261 |
| P261 | 12-000059-00 | Connector, Male, 32 Pin |
| R261 | 39-000257-00 | Resistor, 56K Ohm, ½W, 10% |
| R262 | 39-000130-00 | Resistor, 100K Ohm, ½W, 10% |
| R263 | 39-000117-00 | Res, 1 MEG Ohm ½W, 1% |
| R264 | 39-000132-00 | Res, 499K Ohm, ½W, 1% |
| R265 | 39-000192-00 | Res, 249K Ohm,, ½W, 1% |
| R266 | 39-000286-00 | Res, 124K Ohm, ½W, 1% |
| R267 | 39-000285-00 | Res, 82.5K Ohm, ½W, 1% |
| R268 | 39-000284-00 | Res, 61.9K Ohm, ½W, 1% |
| R269 | 39-000283-00 | Res, 49.9K Ohm, ½W, 1% |
| R270 | 39-000282-00 | Res, 41.2K Ohm, ½W, 1% |
| R271 | 39-000281-00 | Res, 33.2K Ohm, ½W, 1% |
| R272 | 39-000280-00 | Res, 24.9K Ohm, ½W, 1% |
| R273 | 39-000211-00 | Resistor, 10K Ohm, ½W, 1% |
| R274 | 39-000525-00 | Potentiometer, 500 Ohm, 10 Turn |
| R275 | 39-000540-00 | Potentiometer, 500 Ohm, 10 Turn, Rear Shaft |
| S261 | 02-000603-00 | RATE °C/MIN Switch/Resistors (See Detail "A") |
| S262 | 48-000175-00 | MODE Switch, Rotary 4 Pole, 4 Pos |
| TB261 | 51-000021-00 | Terminal Strip, 4 Lug, 4 Pos |
| TB262 | 51-000015-00 | Terminal Strip, 4 Lug |
| TB263 | | Same as TB261 |
| TB264 | 51-000028-00 | Terminal Strip, 3 Lug |
| 1 (SCR262) | 02-000810-00 | Heat Sink Assembly w/SCR 2N3872, 22A, 400V |
| 2 (SCR261) | | Same as 1 |
| 3 (SCR263) | | Same as 1 |
| 4 | 02-000808-00 | Temperature Programmer PC Board Assembly |
| 5 | 02-000800-00 | Isothermal PC Board Assembly |
| 6 | 25-000078-00 | Dial, Multiturn, without Lock |
| 7 | 25-000017-00 | Knob, Skirted, ¼" Shaft |
| 8 | 25-000002-00 | Dial, Multiturn, with Lock |
| 9 | 48-000053-00 | SW, Rotary 1P, 11 PSN |

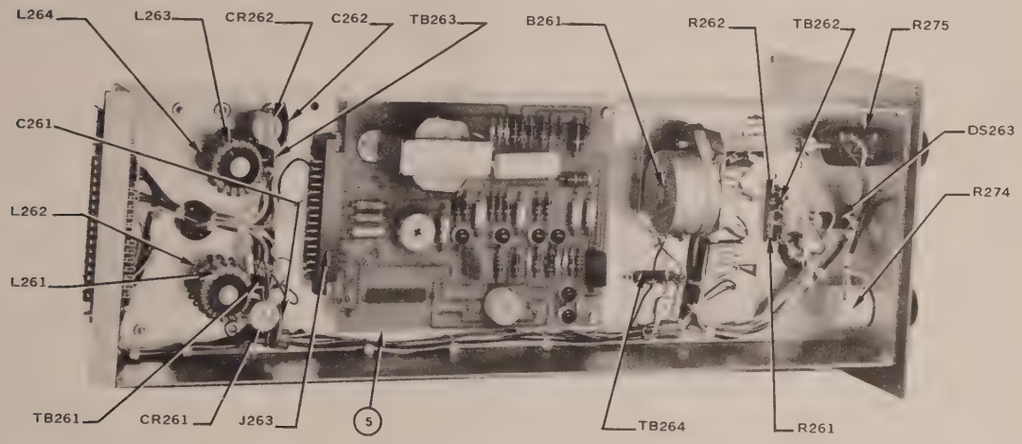


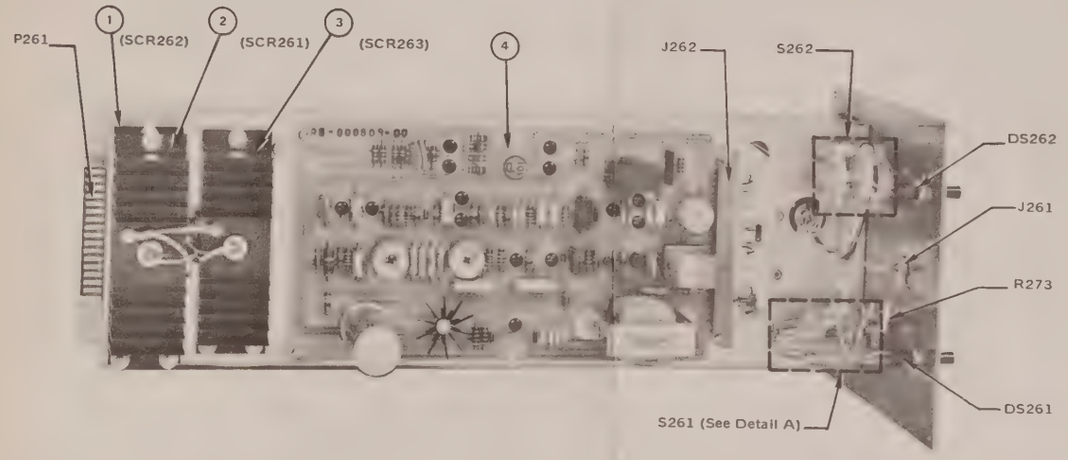

S261
(DETAIL "A")




FRONT VIEW

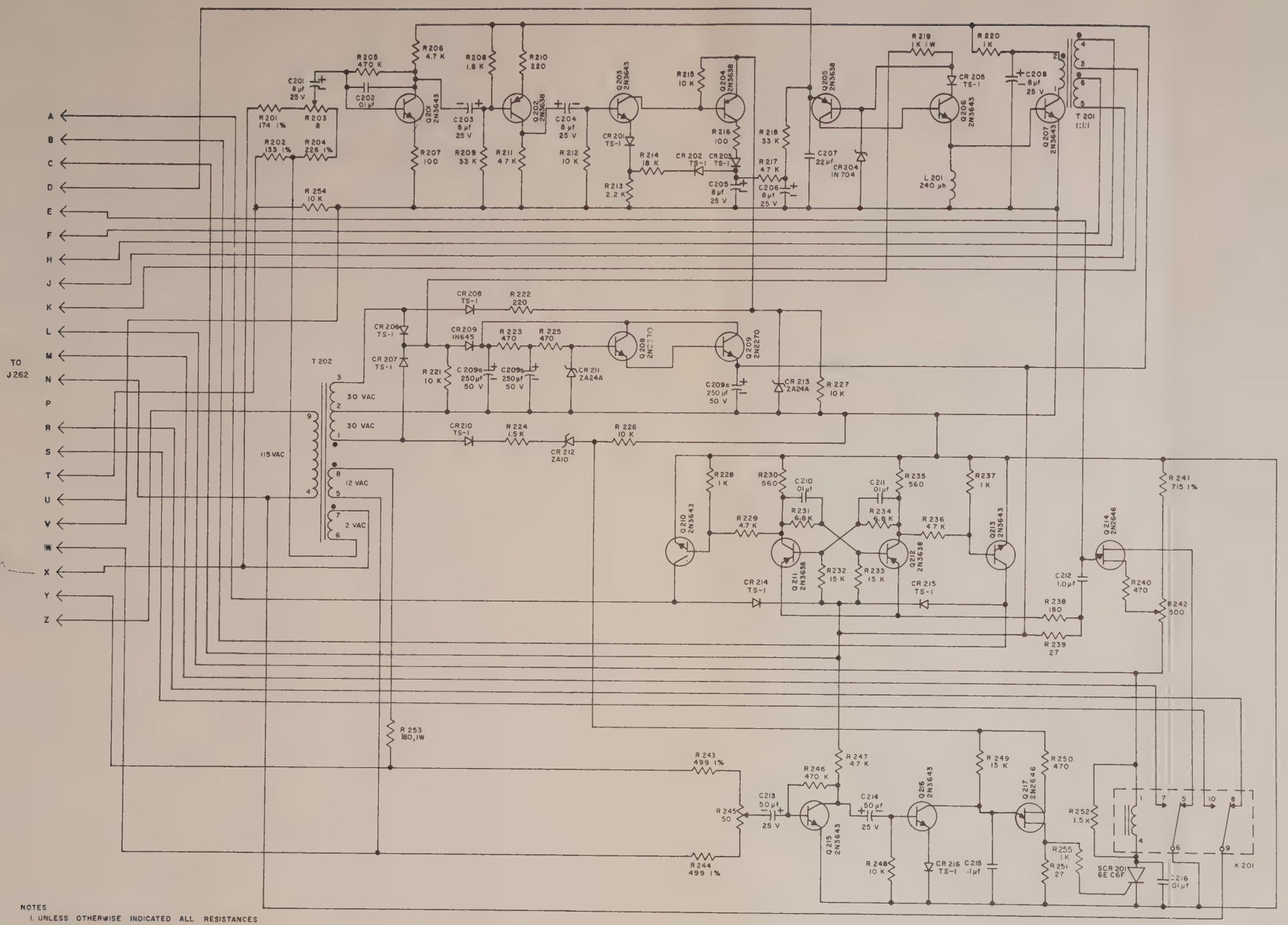



BOTTOM VIEW




TOP VIEW

CHASSIS ELECTRICAL COMPONENTS/HARDWARE
LOCATION DRAWING Issue: New



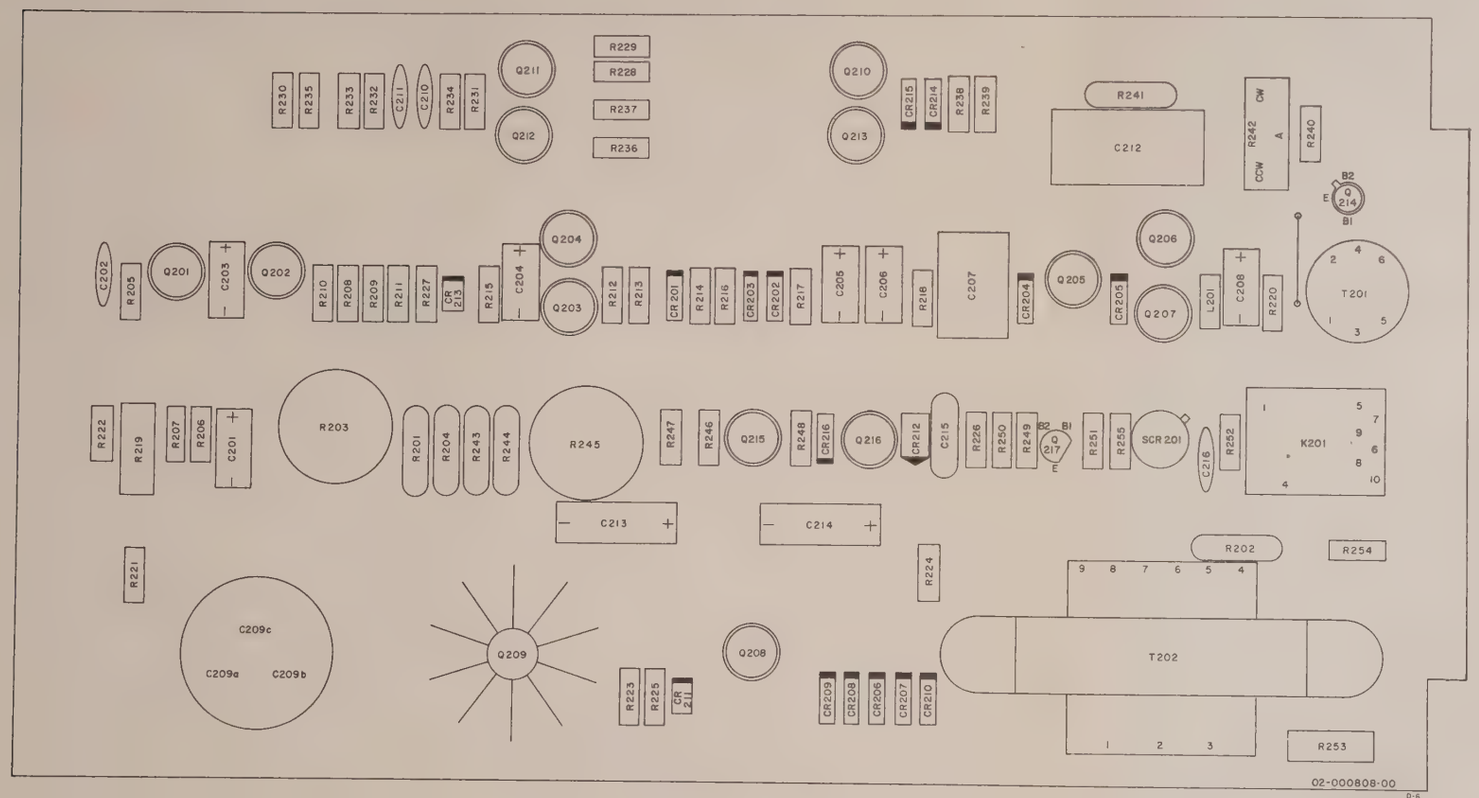

NOTES

1. UNLESS OTHERWISE INDICATED ALL RESISTANCES IN OHMS, 1/2 WATT, 10 %, CARBON.
2. FOR ASS'Y DWG SEE 02 006805-00
3. Q209 REQUIRES A HEAT SINK.

**TEMPERATURE PROGRAMMER PC BOARD SCHEMATIC DIAGRAM**

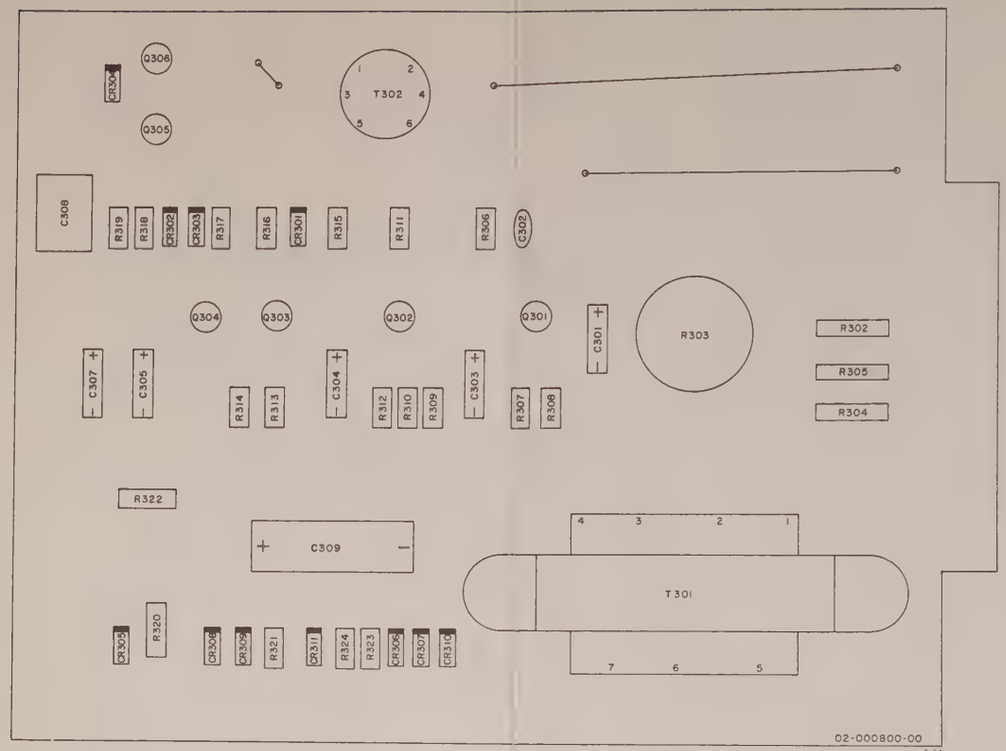
**75-000180-00 Issue: 4**

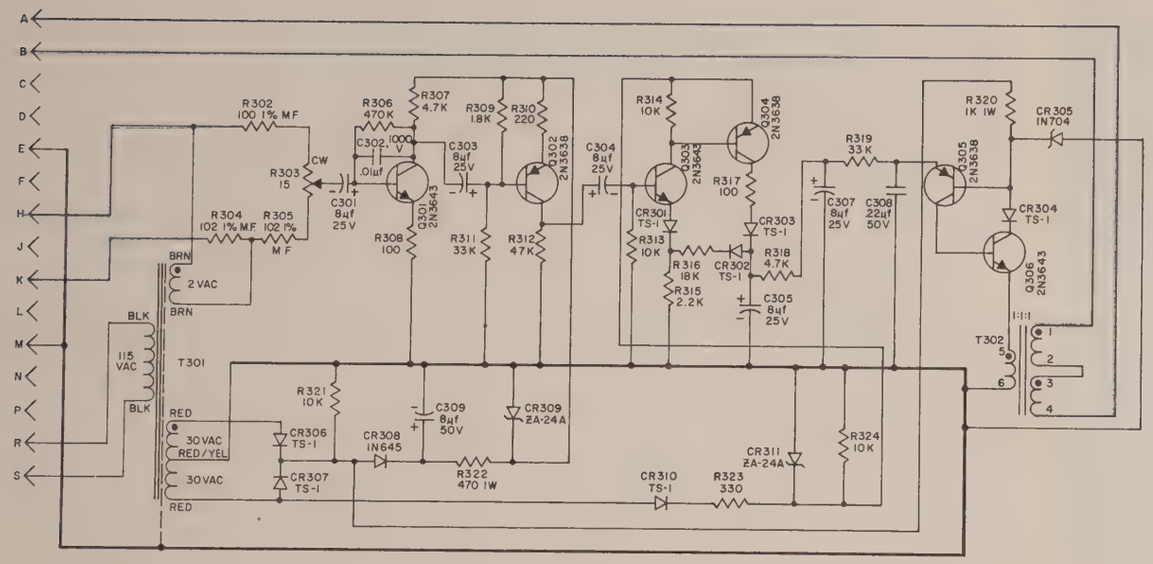
| Parts List/ Schematic Reference Number | VA Part No. | Description |
|---|---|---|
| C201 | 05-000420-00 | Capacitor, Electrolytic, 8 µf, 25V |
| C202 | 05-000004-00 | Capacitor, .01 µf, |
| C203 | 05-000420-00 | Capacitor, Electrolytic, 8 µf, 25V |
| C204 | 05-000420-00 | Capacitor, Electrolytic, 8 µf, 25V |
| C205 | 05-000420-00 | Capacitor, Electrolytic, 8 µf, 25V |
| C206 | 05-000420-00 | Capacitor, Electrolytic, 8 µf, 25V |
| C207 | 05-000241-00 | Capacitor, .22 µf |
| C208 | 05-000420-00 | Capacitor, Electrolytic, 8 µf, 25V |
| C209 a, b, c | 05-000433-00 | Capacitor, Electrolytic, 250 µf, 50V |
| C210 | 05-000004-00 | Capacitor, .01 µf |
| C211 | 05-000004-00 | Capacitor, .01 µf |
| C212 | 05-000242-00 | Capacitor, 1.0 µf |
| C213 | 05-000429-00 | Capacitor, Electrolytic, 50 µf, 25V |
| C214 | 05-000429-00 | Capacitor, Electrolytic, 50 µf, 25V |
| C215 | 05-000236-00 | Capacitor, .1 µf |
| C216 | 05-000004-00 | Capacitor, .01 µf |
| CR201 | 14-000018-00 | Diode, TS-1 |
| CR202 | 14-000018-00 | Diode, TS-1 |
| CR203 | 14-000018-00 | Diode, TS-1 |
| CR204 | 14-000029-00 | Diode, 1N704 |
| CR205 | 14-000018-00 | Diode, TS-1 |
| CR206 | 14-000018-00 | Diode, TS-1 |
| CR207 | 14-000018-00 | Diode, TS-1 |
| CR208 | 14-000018-00 | Diode, TS-1 |
| CR209 | 14-000017-00 | Diode, 1N645 |
| CR210 | 14-000018-00 | Diode, TS-1 |
| CR211 | 14-000016-00 | Diode Zener, ZA24A or Equiv |
| CR212 | 14-000026-00 | Diode Zener, ZA10 |
| CR213 | 14-000016-00 | Diode Zener, ZA24A or Equiv |
| CR214 | 14-000018-00 | Diode, TS-1 |
| CR215 | 14-000018-00 | Diode, TS-1 |
| CR216 | 14-000018-00 | Diode, TS-1 |
| K201 | 48-000527-00 | Relay, TS-154-CC |
| L210 | 53-000401-00 | Coil, 9210-94, Miller, 240 µh |
| Q201 | 14-000508-00 | Transistor, 2N3643 |
| Q202 | 14-000509-00 | Transistor, 2N3638 |
| Q203 | 14-000508-00 | Transistor, 2N3643 |
| Q204 | 14-000509-00 | Transistor, 2N3638 |
| Q205 | 14-000509-00 | Transistor, 2N3638 |
| Q206 | 14-000508-00 | Transistor, 2N3643 |
| Q207 | 14-000508-00 | Transistor, 2N3643 |
| Q208 | 14-000511-00 | Transistor, 2N2270 |
| Q209 | 14-000511-00 | Transistor, 2N2270 |
| Q210 | 14-000508-00 | Transistor, 2N3643 |
| Q211 | 14-000509-00 | Transistor, 2N3638 |
| Q212 | 14-000509-00 | Transistor, 2N3638 |
| Q213 | 14-000508-00 | Transistor, 2N3643 |
| Q214 | 14-000502-00 | Transistor, 2N2646 |
| Q215 | 14-000508-00 | Transistor, 2N3643 |
| Q216 | 14-000508-00 | Transistor, 2N3643 |
| Q217 | 14-000502-00 | Transistor, 2N2646 |
| R201 | 39-000298-00 | Resistor, 174 Ω ½W, 1% |
| R202 | 39-000265-00 | Resistor, 133 Ω ½W, 1% |
| R203 | 39-000538-00 | Resistor, 8 Ω, 2W, 20% |
| R204 | 39-000288-00 | Resistor, 226 Ω, ¼W, 1% |
| R205 | 39-000026-00 | Resistor, 470 K Ω, ½W, 10% |
| R206 | 39-000031-00 | Resistor, 4.7 K Ω, ½W, 10% |
| R207 | 39-000107-00 | Resistor, 100 Ω, ½W, 10% |
| R208 | 39-000146-00 | Resistor, 1.8K Ω, ½W, 10% |
| R209 | 39-000148-00 | Resistor, 33K Ω, ½W, 10% |
| R210 | 39-000240-00 | Resistor, 220 Ω, ½W, 10% |
| R211 | 39-000031-00 | Resistor, 4.7K Ω, ½W, 10% |
| R212 | 39-000147-00 | Resistor, 10K Ω, ½W, 10% |
| R213 | 39-000170-00 | Resistor, 2.2K Ω, ½W, 10% |
| R214 | 39-000229-00 | Resistor, 18K Ω, ½W, 10% |
| R215 | 39-000147-00 | Resistor, 10K Ω, ½W, 10% |
| R216 | 39-000107-00 | Resistor, 100 Ω, ½W, 10% |
| R217 | 39-000031-00 | Resistor, 4.7K Ω, ½W, 10% |
| R218 | 39-000148-00 | Resistor, 33K Ω, ½W, 10% |
| R219 | 39-000004-00 | Resistor, 1K Ω, 1W, 10% |
| R220 | 39-000032-00 | Resistor, 1K Ω, ½W, 10% |
| R221 | 39-000147-00 | Resistor, 10K Ω, ½W, 10% |
| R222 | 39-000240-00 | Resistor, 220 Ω, ½W,10% |
| R223 | 39-000241-00 | Resistor, 470 Ω, ½W, 10% |
| R224 | 39-000134-00 | Resistor, 1.5K Ω, ½W, 10% |
| R225 | 39-000241-00 | Resistor, 470 Ω, ½W, 10% |
| R226 | 39-000147-00 | Resistor, 10K Ω, ½W, 10% |
| R227 | 39-000147-00 | Resistor, 10KΩ, ½W, 10% |
| R228 | 39-000032-00 | Resistor, 1K Ω ½W, 10% |
| R229 | 39-000031-00 | Resistor, 4.7K Ω, ½W, 10% |
| R230 | 39-000137-00 | Resistor, 560 Ω, ½W, 10% |
| R231 | 39-000071-00 | Resistor, 6.8K Ω, ½W, 10% |
| R232 | 39-000231-00 | Resistor, 15K Ω ½W, 10% |
| R233 | 39-000231-00 | Resistor, 15K Ω, ½W, 10% |
| R234 | 39-000071-00 | Resistor, 6.8K Ω, ½W, 10% |
| R235 | 39-000137-00 | Resistor, 560 Ω, ½W, 10% |
| R236 | 39-000031-00 | Resistor, 4.7K Ω, ½W, 10% |
| R237 | 39-000032-00 | Resistor, 1K Ω, ½W, 10% |
| R238 | 39-000302-00 | Resistor, 180 Ω, ½W, 10% |
| R239 | 39-000033-00 | Resistor, 27 Ω, ½W, 10% |
| R240 | 39-000241-00 | Resistor, 470 Ω, ½W, 10% |
| R241 | 39-000056-00 | Resistor, 715 Ω, ½W, 1% |
| R242 | 39-000554-00 | Resistor, 500 Ω, 22 Turn, 2600-P, 1W, 10% |
| R243 | 39-000006-00 | Resistor, 499 Ω, ½W, 1% |
| R244 | 39-000006-00 | Resistor, 499 Ω, ½W, 1% |
| R245 | 39-000553-00 | Resistor, 50 Ω, 2W, 20% |
| R246 | 39-000026-00 | Resistor, 470K Ω, ½W, 10% |
| R247 | 39-000031-00 | Resistor, 4.7K Ω, ½W, 10% |
| R248 | 39-000147-00 | Resistor, 10K Ω, ½W, 10% |
| R249 | 39-000231-00 | Resistor, 15K Ω, ½W, 10% |
| R250 | 39-000241-00 | Resistor, 470 Ω, ½W, 10% |
| R251 | 39-000033-00 | Resistor, 27 Ω, ½W, 10% |
| R252 | 39-000134-00 | Resistor, 1.5K Ω, ½W, 10% |
| R253 | 39-000046-00 | Resistor, 180 Ω, 1W, 10% |
| R254 | 39-000147-00 | Resistor, 10K Ω, ½W, 10% |
| R255 | 39-000032-00 | Resistor, 1K Ω, ½W, 10% |
| SCR201 | 14-000316-00 | SCR, 6EC6F |
| T201 | 53-000024-00 | Transformer Pulse |
| T202 | 53-000032-00 | Transformer, Power |
| | 50-000058-00 | P.C. Board Temp Programmer |
| | 17-000016-00 | Heat Sink Transistor |




**TEMPERATURE PROGRAMMER PC BOARD**
**DRAWING: 02-000808-00 Issue: 7**

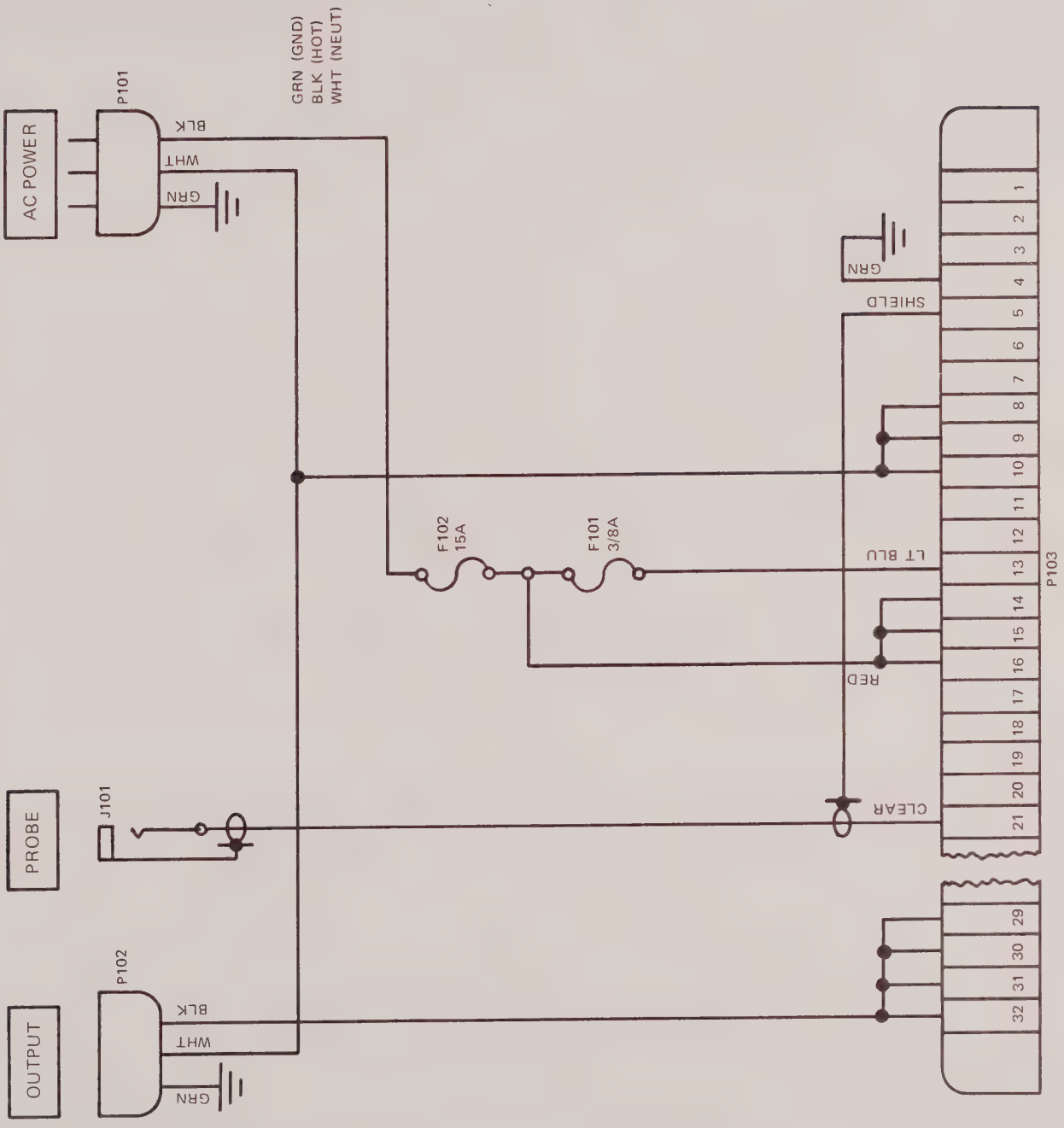
| Parts List/ Schematic Reference Number | VA Part No. | Description |
|---|---|---|
| C301 | 05-000420-00 | Cap., Electrolytic, 8 µf, 25V |
| C302 | 05-000004-00 | Cap., .01 µf, 1000V |
| C303 | | Same as C301 |
| C304 | | Same as C301 |
| C305 | | Same as C301 |
| C307 | | Same as C301 |
| C308 | 05-000241-00 | Cap., .22 µf, 50V |
| C309 | 05-000434-00 | Cap., Electrolytic, 80 µf, 50V |
| CR301 | 14-000018-00 | Diode, TS-1 |
| CR302 | | Same as CR301 |
| CR303 | | Same as CR301 |
| CR304 | | Same as CR301 |
| CR305 | 14-000029-00 | Diode, 1N704 |
| CR306 | | Same as CR301 |
| CR307 | | Same as CR301 |
| CR308 | 14-000017-00 | Diode, 1N645 |
| CR309 | 14-000016-00 | Diode, Zener, 24V, 10%, 1W |
| CR310 | 14-000018-00 | Diode, TS-1 |
| CR311 | | Same as CR309 |
| Q301 | 14-000508-00 | TSTR, NPN, 2N3643 |
| Q302 | 14-000509-00 | TSTR, PNP, 2N3638 |
| Q303 | | Same as Q301 |
| Q304 | | Same as Q302 |
| Q305 | | Same as Q302 |
| Q306 | | Same as Q301 |
| R302 | 39-000092-00 | Res, 1% M.F., 100 Ω, ½W |
| R303 | 39-000543-00 | Pot., 15 Ω, 2W, U-39 |
| R304 | 39-000210-00 | Res, 1% M.F., 102 Ω, ½W |
| R305 | | Same as R304 |
| R306 | 39-000026-00 | Res, 10% Carbon, 470K Ω, ½W |
| R307 | 39-000031-00 | Res, 10% Carbon, 4.7K Ω, ½W |
| R308 | 39-000107-00 | Res, 10% Carbon, 100 Ω, ½W |
| R309 | 39-000146-00 | Res, 10% Carbon, 1.8K Ω, ½W |
| R310 | 39-000240-00 | Res, 10% Carbon, 220 Ω, ½W |
| R311 | 39-000148-00 | Res, 10% Carbon, 33K Ω, ½W |
| R312 | | Same as R307 |
| R313 | 39-000147-00 | Res, 10% Carbon, 10K Ω, ½W |
| R314 | | Same as R313 |
| R315 | 39-000170-00 | Res, 10% Carbon, 2.2K Ω, ½W |
| R316 | 39-000229-00 | Res, 10% Carbon, 18K Ω, ½W |
| R317 | | Same as R308 |
| R318 | | Same as R307 |
| R319 | | Same as R311 |
| R320 | 39-000004-00 | Res, 10% Carbon, 1K Ω, 1W |
| R321 | | Same as R313 |
| R322 | 39-000142-00 | Res, 10% Carbon, 470 Ω, 1W |
| R323 | 39-000070-00 | Res, 10% Carbon, 330 Ω, ½W |
| R324 | | Same as R313 |
| T301 | 53-000025-00 | XFMR, Power, 50/60 Hz |
| T302 | 53-000024-00 | XFMR, Pulse, Sprague 40Z883 |




75-000203-00

**ISOTHERMAL CONTROL PC BOARD DRAWING:**
02-000800-00 Issue: 4
**ISOTHERMAL CONTROL PC BOARD SCHEMATIC DIAGRAM:** 75-000203-00 Issue: New




**MODEL 330 INTERCONNECTION WIRING**
**DIAGRAM: 75-000293-00 Issue: 1**

varian aerograph

2700 mitchell drive/walnut creek/california 94598/415-939-2400
specialists in chromatography and data handling systems

# DUAL/DIFFERENTIAL ELECTROMETER

# INSTRUCTION MANUAL

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Manufactured in the United States of America

PUBLICATION NO. 85-001118-06 PRINTED IN U.S.A. OCTOBER 1970

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DUAL/DIFFERENTIAL ELECTROMETER

# SECTION 1
# INTRODUCTION

## 1.1 GENERAL

The Varian Aerograph™ Dual/Differential Electrometer is designed for use with all Varian Aerograph ionization type detectors, e.g., FID, AFID, $^{3}H$ and $^{63}Ni$. The Electrometer was primarily designed to be installed in the 2740, 2840, and 2860 Series instruments. However, the Electrometer can be purchased as an outboard unit complete with its own cabinet and interconnecting cables.

Basically, the Electrometer converts the small current changes at the output of the ionization detector(s) to representative voltage changes which are capable of driving a pen recorder, integrator, or computer. Besides acting as a current-to-voltage converter, the Electrometer provides the proper interface between the detector(s) and recorder.

Refer to Table 1-1 for specifications on Electrometer.

TABLE 1-1
ELECTROMETER SPECIFICATIONS

**Operating Modes:**
Independent A and B channels; Differential A minus B; B minus A; Simultaneous flame and electron capture (or any two ionization detectors, including FID, AFID, and $^{3}H$ or $^{63}Ni$ electron capture).

**Circuitry:**
Completely Solid State with solid-state preamp utilizing Junction Field Effect Transistors for minimum drift and unsurpassed reliability.

**Sensitivity:**
$1 \times 10^{-12}$ AMPS/MV

**Drift:**
Less than 10 $\mu$V per month long term drift at maximum sensitivity and constant environmental temperature (after 15 minute warm-up and with input shielded).

**Noise**
Less than 10 $\mu$V at maximum sensitivity ($1 \times 10^{-12}$ AMPS/MV).

**Background Suppression:**
$9 \times 10^{-10}$ amp on flame, $9 \times 10^{-8}$ amp on electron capture.

**Dynamic Range:**
$1 \times 10^{5}$ without range change; $1 \times 10^{8}$ with range change.

**Output impedance:**
0 to 5 K ohms.

**Detector Cell Voltage:**
300 Vdc, flame mode; 90 Vdc, electron capture mode.

# SECTION 2
# OPERATION

## 2.1 GENERAL

The Dual/Differential Electrometer converts the small current changes at the output of the ionization detector to representative voltage changes which are capable of driving a pen recorder, integrator or computer. The manner in which the Electrometer performs its function is dependent on front panel controls. Therefore, to familiarize yourself with the location and functional description of the controls on the Electrometer, refer to Figure 2-1 and accompanying description of controls.

FIGURE 2-1 DUAL/DIFFERENTIAL ELECTROMETER

POWER Lamp
Lights when power is on.

A- and B-BUCKING Controls
Adjust recorder pens to zero baseline by bucking background signals from columns and detectors. Maximum counterclockwise rotation sets BUCKING controls to zero.

A- and B-CELL VOLTAGE Switches
OFF: Detector voltage disconnected.
FL: -300V ionization voltage is applied to cathode of FID and/or AFID detectors.
EC: -90V ionization voltage is applied to cathode of $^{3}H$ and/or $^{63}Ni$ detectors.
IGN: Igniting voltage is applied to flame detector (used with FID and AFID detectors).

A- and B-RANGE Selectors
Input attenuators for Channels A and B.
FL: Four positions provide input ranges of $10^{-12}$, $10^{-11}$, $10^{-10}$, and $10^{-9}$ AMPS/MV for FID and AFID detectors.
EC: Two positions provide input ranges of $10^{-10}$ and $10^{-9}$ AMPS/MV for $^{63}Ni$ and $^{3}H$ detectors.
BAL: Permits balancing Electrometer. Set RANGE switch of each unused electrometer channel to BAL position.

A- and B-ATTENUATOR Selectors
Dual-concentric selectors attenuate output of channels A and B individually or simultaneously when interlocked. Attenuators are interlocked by placing both selectors to same position and depressing center selector. Includes shunt "∞" position for zeroing recorder.

A- and B-BALANCE Controls
Initially sets recorder pen of respective channel to zero baseline.

MODE Selector*
A: Permits operation of any one ionization detector when detector leads are connected to A-signal input connector on electrometer.
B: Permits operation of any one ionization detector when detector leads are connected to B-signal input connector on electrometer.
Differential A-B: Permits column A to be used analytically while column B acts as bleed reference.
Differential B-A: Permits column B to be used analytically while column A acts as bleed reference.
FL/FL: Permits simultaneous operation of two FID or AFID detectors. Channel A is used with detector A. Channel B is used with detector B.
FL/EC: Permits simultaneous operation of one FID or AFID detector and one $^{3}H$ or $^{63}Ni$ detector. Channel A is used with A detector (FID or AFID). Channel B is used with B detector ($^{3}H$ or $^{63}Ni$).
Reverse order of detector installation, i.e., Channel A ($^{3}H$ or $^{63}Ni$), etc., is acceptable.

*Electrometer output appears at INTEGRATOR/RECORDER and COMPUTER connectors at rear of basic instrument. Refer to Basic Instrument Manual for specific signal output conditions.

## 2.2 ELECTROMETER OPERATION

The Electrometer must be set up properly to ensure optimum performance from the ionization detector(s) installed in your instrument. To ensure continued optimum performance from the Electrometer, periodic checks and adjustment of electrometer balance should be made.

Balancing and operating the Electrometer with the various ionization detectors requires different settings of the controls. This section of the manual will outline procedures on balancing and operating the Electrometer with various detectors and detector combinations available.

The output signal from the Electrometer is fed through its attenuator into the RECORDER/INTEGRATOR terminal strip and directly into the COMPUTER connectors located at the rear of the basic instrument. Refer to 2700 and 2800 Series Basic Instrument Instruction Manual for details on which terminals to use for obtaining the desired Electrometer output signal.

There are certain procedures that must be followed when balancing and operating your Electrometer with the various detector and detector combinations. These procedures are outlined in Tables 2-1 and 2-2. In all cases, the detector(s) must be installed in the basic instrument, proper gas flow rates established, and the signal and cell voltage cables hooked up to the detector(s) before the Electrometer can be operated. Refer to the Basic Instrument manual and the appropriate detector manual for the proper hook up of detector cables.

### 2.2.1 Electrometer Balancing & Operation with FID and/or AFID Detectors

The FID or AFID detector is typically installed in channel A. A second FID or AFID detector would then be installed in channel B. Balancing of either the FID or AFID detector is the same. If two detectors are installed, i.e., FID/FID, FID/AFID, or AFID/AFID, each channel is still balanced independently. Use controls associated with channel in which detector is installed. Balance and operate Electrometer with FID and/or AFID detector(s) as outlined in Table 2-1.

NOTE

**Channel A will be referred to in Table 2-1 since this channel is typically used whenever there is at least one FID or AFID detector installed in the basic instrument. Since channels A and B are identical, the procedures outlined apply to both channels.**

### 2.2.2 Electrometer Balancing & Operation with $^{3}H$ and/or $^{63}Ni$ Detectors

The $^{3}H$ or $^{63}Ni$ detector is typically installed in channel B. A second $^{3}H$ or $^{63}Ni$ or possibly an FID or AFID would then be installed in channel A. Balancing procedures for either the $^{3}H$ or $^{63}Ni$ detectors are the same. If two detectors are installed, i.e., $^{3}H/^{3}H$, $^{3}H/^{63}Ni$, or $^{63}Ni/^{63}Ni$, each channel is still balanced independently. Use controls associated with channel in which detector is installed. Operation of a FID or AFID/$^{3}H$ or $^{63}Ni$ detector combinations will be discussed in paragraph 2.2.3. Balance and operate Electrometer with $^{3}H$ and/or $^{63}Ni$ detector(s) as outlined in Table 2-2.

NOTE

**Channel B, i.e., the EC position of the FL-EC mode switch, will be referred to in Table 2-2 since this channel is typically used whenever there is at least one $^{3}H$ or $^{63}Ni$ detector installed in the basic instrument. Since both channels are identical, the procedures outlined apply to both channels.**

### 2.2.3 Electrometer Operation with Combination of FID and EC Detectors

Electrometer operation with detector combinations such as FID or AFID/$^{3}H$ or $^{63}Ni$ is straight forward. The FID or AFID detector should be installed in channel A and the $^{3}H$ or $^{63}Ni$ detector installed in channel B. Each Electrometer channel should be set up with appropriate detector for single channel operation as outlined in Tables 2-1 and 2-2. The appropriate MODE selector position for two channel FID or AFID/$^{3}H$ or $^{63}Ni$ operation is FL-EC.

TABLE 2-1 ELECTROMETER BALANCING AND OPERATION WITH FID and/or AFID DETECTORS

| | BALANCE AND OPERATE ELECTROMETER AS OUTLINED IN FOLLOWING STEPS | MODE SWITCH SETTING | CELL VOLTAGE SWITCH SETTING CH A/CH B | RANGE SWITCH SETTING CH A/CH B | ATTENUATOR SWITCH SETTING CH A/CH B | BALANCE CONTROL SETTING CH A/CH B | BUCKING CONTROL SETTING CH A/CH B | ADDITIONAL COMMENTS AND NOTES |
|---|---|---|---|---|---|---|---|---|
| BALANCING AND FLAME IGNITION | STEP (1) | A | OFF/OFF | BAL/BAL | ∞/∞ positions See comments. | ——— | ——— | Adjust pen recorder for mechanical and electrical zero. |
| | STEP (2) | A | OFF/OFF | BAL/BAL | 1X/1X See comments | Adjust Channel A control to zero recorder pen | ——— | ATTENUATOR control is dual concentric. The inner control knob is for channel A and the outer control knob is for channel B. The A-control knob should be pulled out when either channel is operated independently. |
| | STEP (3) | A | FL/OFF | FL/$10^{-11}$/BAL | 1X/1X | ——— | Set Ch A to maximum counter-clockwise position | |
| | STEP (4) | A | FL (IGN)/OFF See comments | FL/$10^{-11}$/BAL | 1X/1X | ——— | Adjust Ch A control to zero recorder pen | Ignite flame by holding A-cell voltage switch to IGN position for about 3 to 5 seconds. When released, spring loaded switch will return to FL position. A large upscale deflection of recorder pen will indicate flame ignition. If flame fails to ignite, remove tower cap and note if ignition coil glows when using IGN position. If ignitor coil glows but flame does not ignite, try increasing $H_2$ pressure to detector momentarily and/or turning off carrier gas momentarily. Note that flame cannot be seen; therefore, visual inspection of detector will not confirm flame ignition. Flame ignition can be tested by passing a small piece of cotton (attached to a stick and dipped in alcohol) over the flame tower. If flame is lit, cotton will burst into flame. |
| | STEP (5) | | | ——— SEE COMMENTS ——— | | | | If second detector is installed, repeat first 4 steps using B channel controls. |
| OPERATION | STEP (6) Single Channel Operation | A or B | FL/OFF (Ch A) or OFF/FL (Ch B) | FL/$10^{-9}$ to $10^{-12}$ (either channel) See comments | 1X to 1024X (either channel) See comments | ——— | ——— | The RANGE switch should be adjusted so that the dynamic range of the GC peaks can be handled without saturating the electrometer amplifier. With proper adjustment of the RANGE switch, the peaks can be kept on scale by adjustment of the ATTENUATOR switch. A quick way to make sure the electrometer input amplifier is not saturated is to set ATTENUATOR switch to 1024X. If none of the peaks go off scale, independent of RANGE switch settings, the input amplifier is not being saturated. |
| | Two (independent) channel operation | FL-FL | FL/FL | FL/$10^{-9}$ to $10^{-12}$ (ADJUST RANGE A & B switches independently). See comments. | 1X to 1024X (ADJUST ATTENUATOR A & B switches independently). See comments. | ——— | ——— | The balance of either electrometer channel can be checked at any time by setting the appropriate RANGE switch to BAL and the ATTENUATOR switch to 1X. If recorder pen move off scale, rezero with appropriate BALANCE control, then return RANGE and ATTENUATOR controls to their previous positions. |
| | Differential Operation | A-B or B-A See comments | FL/FL | FL/$10^{-9}$ to $10^{-12}$ (Set both RANGE A & B switches to same positions). | 1X to 1024X (Set ATTENUATOR A&B switches to same position and interlock switches by pressing A switch inward). See comments | ——— | Readjust BUCKING A or B control to zero recorder pen. | When differential A-B mode of operation is selected (sample injected into A system), readjust BUCKING B control to rezero recorder pen. When differential B-A mode is selected (sample injected into B system), readjust BUCKING A control to rezero recorder pen. |

TABLE 2-2 ELECTROMETER BALANCING AND OPERATION WITH $^{3}H$ and $^{63}Ni$ ELECTRON CAPTURE DETECTORS

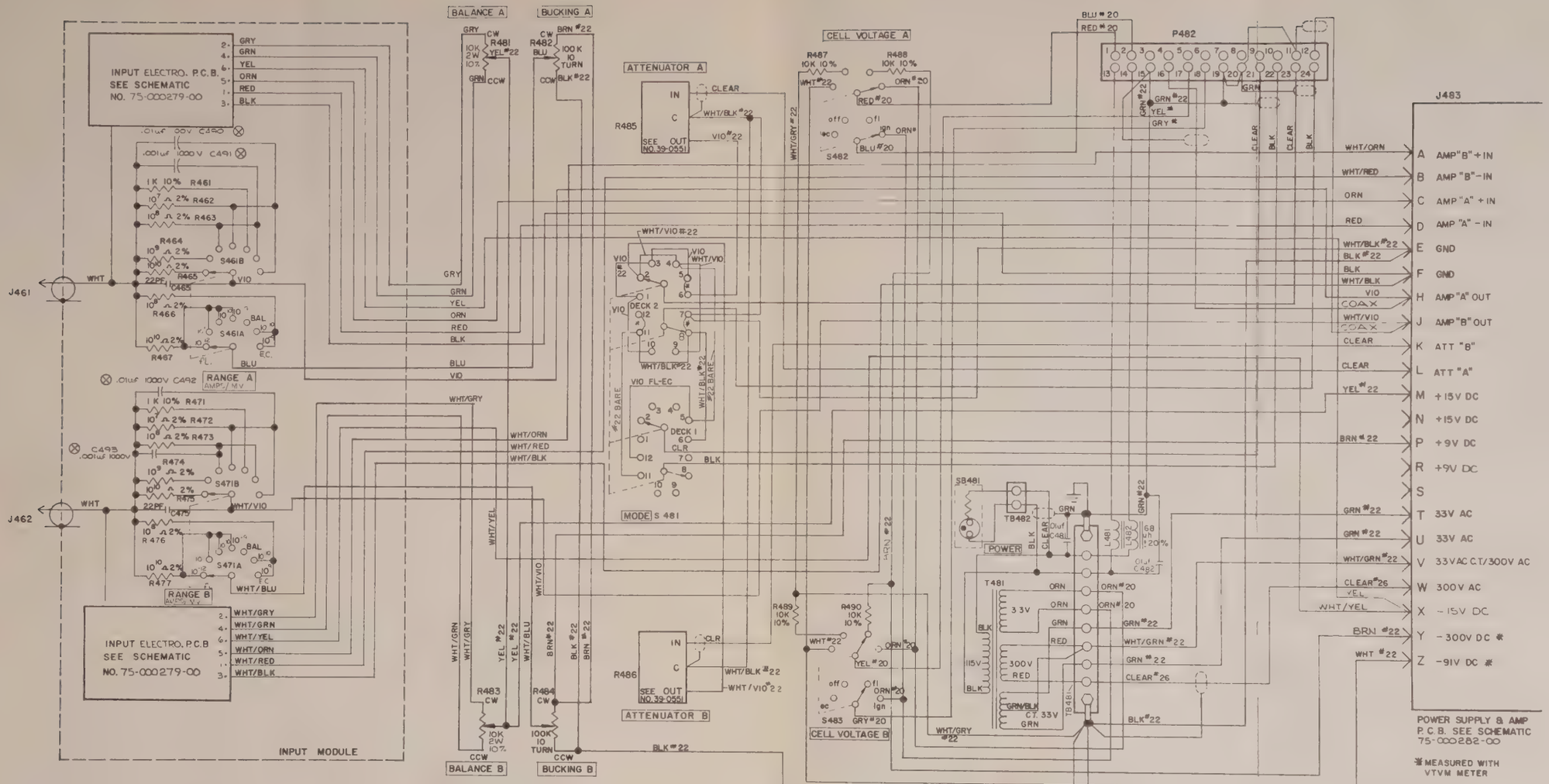
| | BALANCE AND OPERATE ELECTROMETER AS OUTLINED IN FOLLOWING STEPS | MODE SWITCH SETTING CH A/CH B | CELL VOLTAGE SWITCH SETTING CH A/CH B | RANGE SWITCH SETTING CH A/CH B | ATTENUATOR SWITCH SETTING CH A/CH B | BALANCE CONTROL SETTING CH A/CH B | BUCKING CONTROL SETTING CH A/CH B | ADDITIONAL COMMENTS AND NOTES |
|---|---|---|---|---|---|---|---|---|
| — BALANCING — | STEP (1) | FL-EC | OFF/OFF | BAL/BAL | ∞/∞ positions See comments | ——— | ——— | Adjust pen recorder for mechanical and electrical zero. |
| | STEP (2) | FL-EC | OFF/OFF | BAL/BAL | 1X/1X See comments. | Adjust B channel control to zero recorder pen | ——— | ATTENUATOR control is dual concentric. The inner control knob is for channel A and the outer central knob is for channel B. The A-control knob should be pulled out when either channel is operated independently. |
| | STEP (3) | | | ——— SEE COMMENTS ——— | | | | If second detector is installed in channel A, repeat first 2 steps using A-channel controls. Note the A-recorder leads must be reversed when using an electron capture detector in channel A. |
| — STANDING CURRENT CHECK — | STEP (4) Make sure electrometer is balanced as outlined in steps (1) through (4), then check standing current of EC detector as follows: | FL-EC | OFF/EC | BAL/EC $10^{-9}$ | 1X/32X ($^{3}H$) 1X/2X ($^{63}Ni$) | ——— | Zero recorder pen with BUCKING B control | |
| | STEP (5) | FL-EC | OFF/OFF | | ——— SEE COMMENTS ——— | | | Observe number of divisions pen deflects and multiply this number times the ATTENUATOR setting of 32X ($^{3}H$ detector) or 2X $^{63}Ni$ detector) and the RANGE setting of $10^{-9}$A. The recorder pen should deflect at least 40 divisions ($^{3}H$ detector) or 50 divisions ($^{63}Ni$ detector) of full scale (1 mV recorder) with new detector. Standing Current for $^{3}H$ detector = 32X · $10^{-9}$A · 40 divisions = 1.28 X $10^{-8}$A. Standing Current for $^{63}Ni$ detector = 2X · $10^{-9}$A · 50 divisions = 1.00 X $10^{-9}$A. |
| — OPERATION — | STEP (6) Single channel operation | FL-EC | OFF/EC | BAL/EC $10^{-9}$ or $10^{-10}$. See comments. | 1X/1X to 1024X See Comments | ——— | Zero recorder pen with BUCKING B control | The RANGE switch should be adjusted so that the dynamic range of the GC peaks can be handled without saturating the electrometer amplifier. With proper adjustment of the RANGE switch, the peaks can be kept on scale by adjustment of the ATTENUATOR switch. A quick way to make sure the electrometer input amplifier is not saturated is to set ATTENUATOR switch to 1024X. If none of the peaks go off scale, independent of RANGE switch settings, the input amplifier is not being saturated. |
| | Two (independent) channel operation | FL-EC see comment on Step (4) | EC/EC | EC $10^{-9}$ or $10^{-10}$/ EC $10^{-9}$ or $10^{-10}$ (adjust RANGE A & B switches independently). See comments. | 1X to 1024X/1X to 1024X (adjust ATTENUATOR A & B switches independently). See comments. | ——— | Zero both recorder pens with appropriate BUCKING controls | The balance of either electrometer channel can be checked at anytime by setting the appropriate RANGE switch to BAL and the ATTENUATOR switch to 1X. If recorder pen moves off scale, rezero with appropriate BALANCE control, then return RANGE and ATTENUATOR controls to their previous positions. |

# SECTION 3
# ILLUSTRATED PARTS LISTS/SCHEMATICS

## 3.1 GENERAL

This section of the manual contains illustrated parts lists and schematic diagrams for the Dual/Differential Electrometer. Listed below in the order they appear are the titles and drawing numbers (where applicable) of all illustrated parts lists and schematic diagrams.


| VA TITLE | DWG. NO. | PAGE NO. |
|---|---|---|
| Interconnection Schematic Diagram | 75-000281-00 Issue 3 | 3-2 |
| Chassis Electrical Component/Hardware Location Drawing | ——— | 3-3 |
| Power Supply & Amplifier Schematic Diagram | 75-000282-00 Issue 1 | 3-4 |
| Power Supply & Amplifier PC Board Drawing | 02-001138-00 Issue 3 | 3-5 |
| JFET Input Schematic Diagram | 75-000279-00 Issue 2 | 3-6 |
| JFET Input PC Board Drawing | 02-001131-00 Issue 2 | 3-6 |



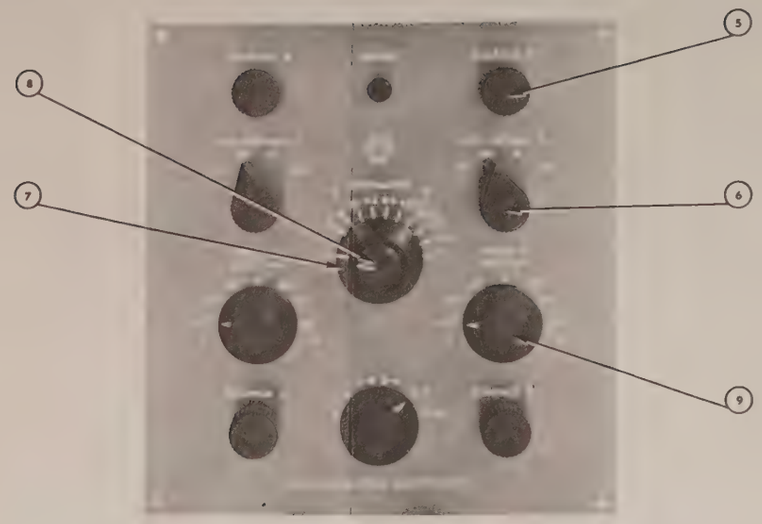

NOTES

1. ALL WIRES #24 UNLESS INDICATED.
2. ALL SWITCHES SHOWN FRONT VIEW.
3. ⏚ CHASSIS GROUND
4. LUGS INTERNALLY JUMPED. (*)
5. UNLESS OTHERWISE INDICATED ALL D.C. VOLTAGES MEASURED WITH 20K OHMS PER VOLT METER TO GROUND AND ALL A.C VOLTAGES MEASURED WITH 5K OHMS PER VOLT METER.
6. ALL POTENTIOMETERS ARE 3 WATT, 3%, WIRE WOUND UNLESS OTHERWISE NOTED.
7. THE SYMBOL ⊗ INDICATES COMPONENTS USED ONLY ON MODELS 00-173210-00 AND 00-173220-00.

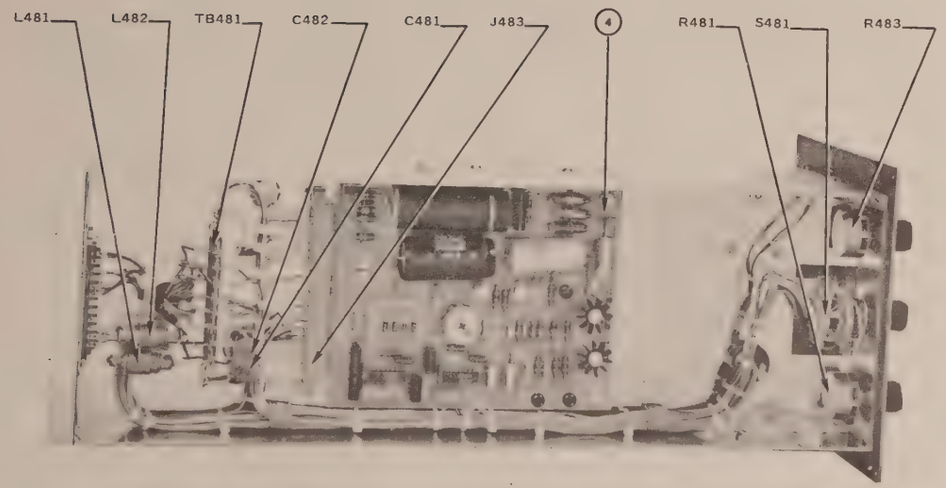
**ELECTROMETER INTERCONNECTION SCHEMATIC DIAGRAM**
Schematic: 75-000281-00 Issue: 3

| Parts List/ Schematic Reference Number | VA Part No. | Description |
|---|---|---|
| C481 | 05-000004-00 | Capacitor, .01 μfd, 1000 WVDC |
| C482 | | Same as C481 |
| J461 | 12-000041-00 | Connector, BNC, Female, UG1094 |
| J462 | | Same as J461 |
| J483 | 12-000139-00 | Connector, PC Board, 22 Pin |
| L481 | 53-000402-00 | Choke, 68 μh, 2A |
| L482 | | Same as L481 |
| P482 | 12-000056-00 | Connector, Male, 24 Pin |
| R481 | 39-000524-00 | Potentiometer, 10K Ω, 2W, 10% |
| R482 | 39-000512-00 | Potentiometer, 100K Ω, 3W, 3% |
| R483 | | Same as R481 |
| R484 | | Same as R482 |
| R485 | 39-000551-00 | Attenuator, Dual, 5K Ω, (Back Section) |
| R486 | | Part of R485 (Front Section) |
| S461 | 01-000540-00 | Range Switch Assembly w/resistors |
| S471 | | Same as S461 |

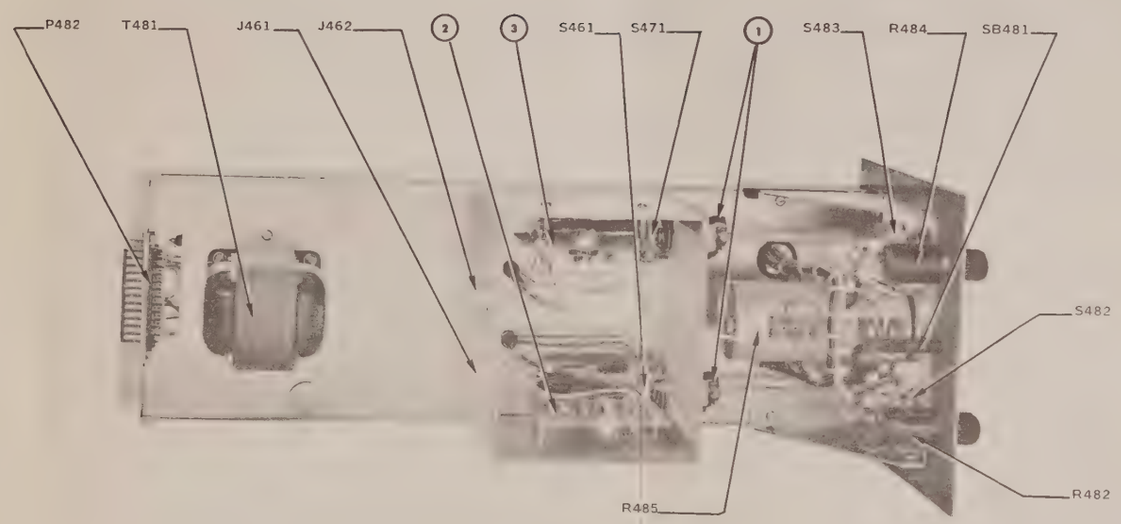
| Parts List/ Schematic Reference Number | VA Part No. | Description |
|---|---|---|
| TB481 | 51-000001-00 | Terminal Strip, 8 Lugs Insulated |
| S481 | 01-000422-00 | Mode Switch Subassembly |
| S482 | 48-000123-00 | Rotary Switch, 2 Pole, 4 Position |
| S483 | | Same as S482 |
| SB481 | 35-000023-00 | Lamp, Red, Neon w/Res |
| T481 | 53-000036-00 | Power Transformer, 115 Vac, 50/60 $H_z$ |
| TB482 | 51-000016-00 | Terminal Strip, 2 Lug Insulated |
| 1 | 16-000128-00 | Coupling, ¼ x ¼ in |
| 2 | 02-001131-00 | JFET Input Module PC Board |
| 3 | | Same as 2 |
| 4 | 02-001138-00 | Power Supply & Amp. PC Board |
| 5 | 25-000018-00 | KNOB, #70-2-2G |
| 6 | 25-000021-00 | KNOB, TEARDROP, #705-2G |
| 7 | 02-000681-00 | Knob, Outer |
| 8 | 25-000075-00 | Knob, Inner |
| 9 | 25-000017-00 | Knob, Skirted #70-3-2G |




Front View

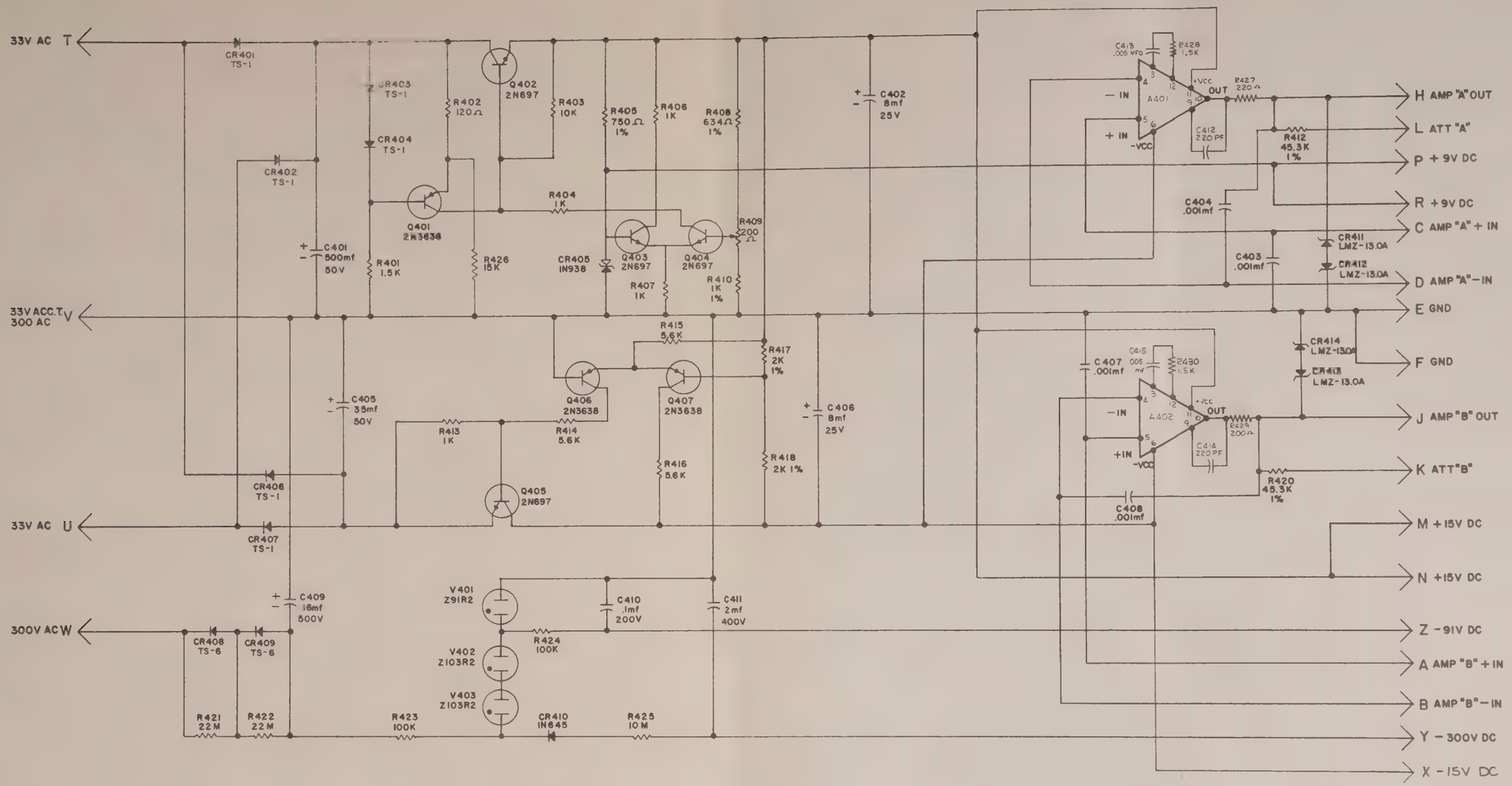



Bottom View




Top View

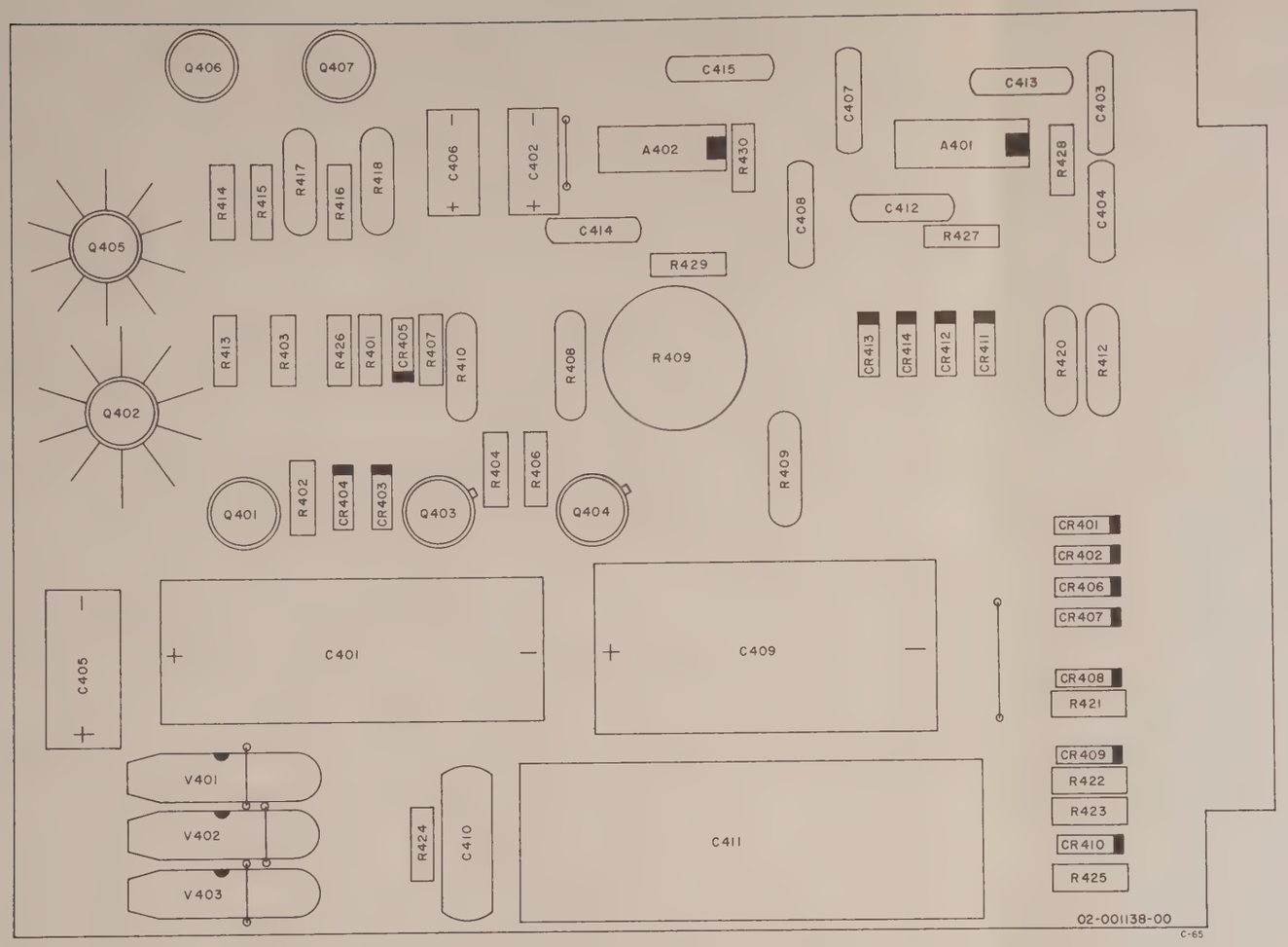
**CHASSIS ELECTRICAL COMPONENT/ HARDWARE LOCATION DRAWING**
Issue: New




NOTES

1. ALL RESISTORS ARE 1/2 W 10% UNLESS INDICATED.
[1] 2. REFERENCE ASS'Y 02-001138-00, ISSUE "2.

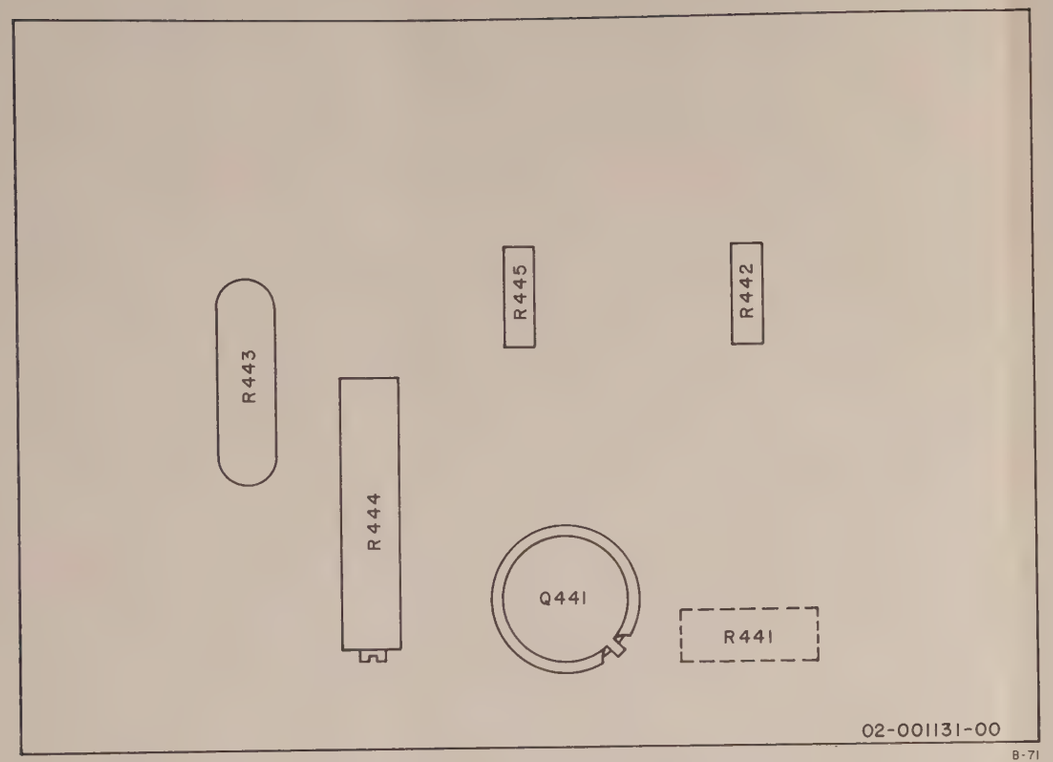
**POWER SUPPLY & AMPLIFIER**
**SCHEMATIC DIAGRAM**
Schematic: 75-000282-00
Issue: 1

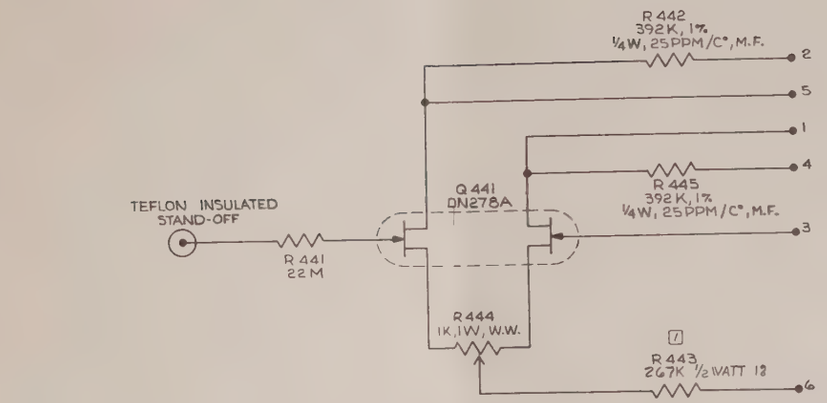



| Parts List/ Schematic Reference Number | VA Part No. | Description |
|---|---|---|
| A401 & A402 | 14-000567-00 | Integrated Ckt. |
| C401 | 05-000417-00 | Cap, 500 MFD, 50V |
| C402 | 05-000420-00 | Cap, 8 MFD, 25V |
| C403 & C404 | 05-000239-00 | Cap, .001 MFD, 600V |
| C405 | 05-000238-00 | Cap, 35 MFD, 50V |
| C406 | 05-000420-00 | Cap, 8 MFD, 25V |
| C407 & C408 | 05-000239-00 | Cap, .001 MFD, 600V |
| C409 | 05-000439-00 | Cap, 16 MFD, 500V |
| C410 | 05-000240-00 | Cap, .1 MFD, 200V |
| C411 | 05-000218-00 | Cap, 2 MFD, 400V |
| C412 | 05-000012-00 | Cap, 220 PFD |
| C413 | 05-000221-00 | Cap, .005 MFD |
| C414 | 05-000012-00 | Cap, 220 PFD |
| C415 | 05-000221-00 | Cap, .005 MFD |
| CR401 thru CR403 | 14-000018-00 | Diode TS-1 |
| CR404 | 14-000018-00 | Diode TS-1 |
| CR405 | 14-000024-00 | Diode IN938 |
| CR406 & CR407 | 14-000018-00 | Diode TS-1 |
| CR408 & CR409 | 14-000005-00 | Diode TS-6 |
| CR410 | 14-000017-00 | Diode IN645 |
| CR411 thru CR414 | 14-000025-00 | Diode LMZ-13.0A |
| Q401 | 14-000509-00 | TSTR, 2N3638 |
| Q402 thru Q405 | 14-000504-00 | TSTR, 2N697 |
| Q406 & Q407 | 14-000509-00 | TSTR, 2N3638 |
| R401 | 39-000134-00 | Res, 1.5K, ½W, 10% |
| R402 | 39-000274-00 | Res, 120 Ω, ½W, 10% |
| R403 | 39-000147-00 | Res, 10K, ½W, 10% |
| R404 | 39-000032-00 | Res, 1K, ½W, 10% |
| R405 | 39-000275-00 | Res, 750 Ω, ½W, 1% |
| R406 & R407 | 39-000032-00 | Res, 1K, ½W, 10% |
| R408 | 39-000078-00 | Res, 634 Ω, ½W, 1% |
| R409 | 39-000541-00 | Pot, 200 Ω, 2W, 20% |
| R410 | 39-000007-00 | Res, 1K, ½W, 1% |
| R412 | 39-000276-00 | Res, 45.3K, ½W, 1% |
| R413 | 39-000032-00 | Res, 1K, ½W, 10% |
| R414 thru R416 | 39-000268-00 | Res, 5.6K, ½W, 10% |
| R417 & R418 | 39-000008-00 | Res, 2K, ½W, 1% |
| R420 | 39-000276-00 | Res, 45.3K, ½W, 1% |
| R421 & R422 | 39-000102-00 | Res, 22M, ½W, 10% |
| R423 & R424 | 39-000130-00 | Res, 100K, ½W, 10% |
| R425 | 39-000149-00 | Res, 10M, ½W, 10% |
| R426 | 39-000231-00 | Res, 15K, ½W, 10% |
| R427 | 39-000240-00 | Res, 220 Ω, ½W, 10% |
| R428 | 39-000134-00 | Res, 1.5K, ½W, 10% |
| R429 | 39-000240-00 | Res, 220 Ω, ½W, 10% |
| R430 | 39-000134-00 | Res, 1.5K, ½W, 10% |
| V401 | 56-000029-00 | Tube, Z91R2 |
| V402 & V403 | 56-000031-00 | Tube, Z103R2 |

**POWER SUPPLY & AMPLIFIER P.C. BOARD**
Assembly Drawing: 02-001138-00 Issue: 3

| Parts List/ Schematic Reference Number | VA Part No. | Description |
|---|---|---|
| Q441 | 14-000563-00 | Transistor, JFET, DN278A |
| | 44-000037-00 | Socket, 6 Pin, Transistor, MTG. |
| R441 | 39-000102-00 | Resistor, 22 Meg, ½W, 10% |
| R442 | 39-000482-00 | Resistor, 392 K, ¼W, 1% |
| R443 | 39-000790-00 | Resistor, 267 K, ½W, 1% MF |
| R444 | 39-000532-00 | Potentiometer, 1K, 1W, 10% |
| R445 | | Same as R442 |
| | 12-000202-00 | Terminal, Insulated Feed Thru |




NOTES:
1. ALL RESISTORS ½ WATT, 10%, CARBON, UNLESS OTHERWISE NOTED.
2. FOR P.C. BOARD ASS'Y SEE 02-001131-00.

**JFET INPUT PC BOARD**
Schematic Diagram: 75-000279-00 Issue: 2
Assembly Drawing: 02-001131-00 Issue: 4

varian aerograph

2700 mitchell drive/walnut creek/california 94598/415-939-2400
specialists in chromatography and data handling systems

# ALKALI FLAME IONIZATION DETECTOR

# INSTRUCTION MANUAL

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PUBLICATION NO. 85-001118-11

## SAFETY PRECAUTIONS AND OPERATING INSTRUCTIONS

This instruction manual is provided to help you establish operating conditions which will permit efficient use of your equipment. You will notice that the manual describes special considerations and precautions in the form of notes, cautions, and warnings, that not only aid you in obtaining the best performance from your equipment but in calling to your attention potential hazards (e.g., electrical, mechanical, etc.) that might cause personal injury, and how to avoid these hazards. It is important, therefore, that you operate your equipment in accordance with this instruction manual and additional information which may be provided by Varian Instrument Division, Aerograph® Products. Address any questions regarding the safe and proper use of your instrument to:

Varian Instrument Division, Aerograph® Products
611 Hansen Way
Palo Alto, California 94303

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## SPARE PARTS AVAILABILITY

It is the policy of the Varian Instrument Division to provide operational spare parts for any instrument and major accessory for a period of five (5) years after shipment of the final production run of that instrument. Spare parts will be available after this five (5) year period but on an "as available" basis.

Operational spare parts are defined as those individual electrical or mechanical parts that are susceptible to failure during their normal operation. Examples include resistors, inductors, capacitors, semi-conductor devices of all kinds, relays, lamps, temperature probes, detector elements, motors, etc.

Sheet metal parts, structural members or assemblies, and castings, printed circuit cards, and functional modules are normally capable of being rebuilt to like-new condition throughout their useful life and therefore will be supplied on an "as available" basis only after the final production run of the instrument.

The repair and/or reconditioning of instruments and accessories is handled on a time and materials basis after expiration of the Warranty period. Please contact your nearest Varian Instrument Division Sales and Service Office for a quotation.

6/20/1972

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# SECTION 1
# INTRODUCTION

## 1.1 GENERAL

The Varian Aerograph™ Alkali Flame Ionization Detector (AFID) is selectively sensitive to phosphorus and nitrogen-containing compounds. Using a rubidium sulfate flame tip and optimum flow conditions, phosphorus and nitrogen-containing compounds can be detected at the picogram level with a selectivity of 10,000 to 1 over normal hydrocarbons. Replacing the former Varian Aerograph Phosphorus Detector, the AFID provides stability and reliability approaching that of the standard Flame Ionization Detector (FID). Although a theoretical understanding of the ionization process in the alkali flame is not clearly understood, the detector will meet most application requirements by following the installation and operating instructions outlined in this manual.

## 1.2 SPECIFICATIONS

**Sensitivity**
Phosphorus-Containing Compounds:
$3 \times 10^{-14}$ g/sec
Nitrogen-Containing Compounds:
$\approx 1 \times 10^{-12}$ g/sec

**Linearity**
1000 fold

**Background Current**
$\approx 3 \times 10^{-11}$ A

**Noise Level**
$\approx 10^{-13}$ A peak-to-peak

**Ionization Source**
Reducing Hydrogen Flame

**Upper Limit Temperature**
Undetermined; estimated at 275-300°C.

**Polarizing Voltage**
-300 V

**Specificity**
10,000 to 1; greater when detector is tuned for a specific analysis.

# SECTION 2
# INSTALLATION

## 2.1 GENERAL

The Alkali Flame Ionization Detector (AFID) can be installed in any of the following instruments: 1200, 1400, 1700, 1800, 2100, 2700, and 2800 Series. The AFID is purchased as a kit and includes all necessary installation hardware. Table 2-1 identifies the proper Kit Number for each instrument series.

TABLE 2-1
KIT NUMBER/INSTRUMENT IDENTIFICATION

| Kit Number | Instrument Series |
|---|---|
| 02-001352-00 | 1200 |
| 02-001351-00 | 1400 |
| 02-001351-00 | 1700 |
| 02-001351-00 | 1800 |
| 02-001352-00 | 2100 |
| 02-001351-00 | 2700 |
| 02-001351-00 | 2800 |

## 2.2 UNPACKING

When unpacking the AFID Kit, carefully check to ensure that all components ordered have been supplied. Refer to the accessory packing list and the installation instructions in this manual to identify the parts. If there are any discrepancies, notify Varian Aerograph.

NOTE

**If any damage is found, immediately notify the Carrier and Varian Aerograph. Do not return the Kit to the factory without written authorization from Varian Aerograph. Include a copy of the authorization with the Kit when returning Kit to the factory.**

## 2.3 LOCATION

The instrument in which the AFID is installed should be located where it will not be subjected to abrupt temperature changes, excessive vibration, or strong air currents (such as under a heater or air conditioning vent). Ensure adequate space is available for the necessary gas supplies.

## 2.4 GAS CONNECTIONS

Refer to the following paragraphs for gas supplies and controls required to operate the AFID.

NOTE

**Contact your Varian Aerograph Sales Representative for flow measuring and flow control accessories required to install your gas supplies.**

### 2.4.1 Carrier Gas Supply

The required carrier gas supply plumbing is illustrated in Figure 2-1. All of the components shown in Figure 2-1 are necessary except for the flow diverter. However, the flow diverter does allow the carrier gas to be monitored without disconnecting instrument plumbing. The components comprising the carrier gas supply are described in the following paragraphs.

- **Gas Supply**

The gas supply is a standard compressed gas cylinder. Helium or nitrogen can be used for carrier gas although helium is preferred.

- **Two-Stage Pressure Regulator**

The two-stage pressure regulator monitors the gas cylinder pressure and regulates the second stage pressure. Set the second stage pressure for 60 to 100 psi.

- **Shut-Off Valve**

The shut-off valve is used to turn the carrier gas on and off as required. The shut-off valve eliminates the need for using the flow controller valve as an on-and-off valve which damages the controller valve seat.

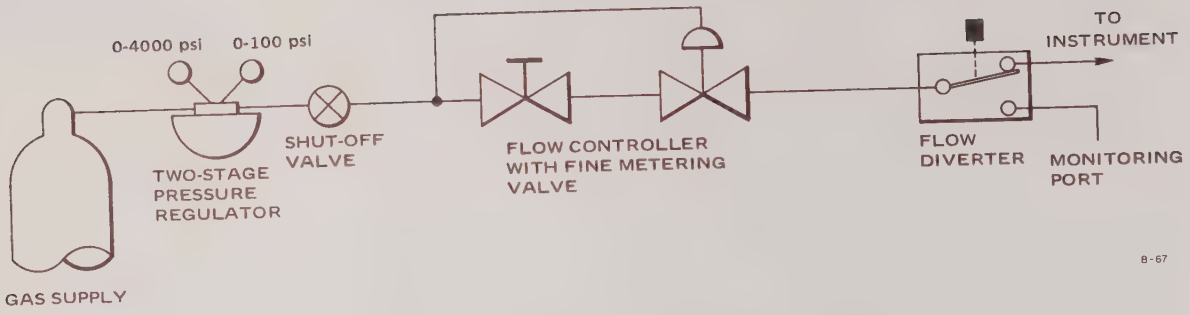



FIGURE 2-1 AFID CARRIER GAS SUPPLY PLUMBING DIAGRAM

• **Flow Controller**
The flow controller maintains a constant gas flow through the column as the column head pressure varies. The desired carrier gas flow rate is set with the fine metering valve and then maintained by the flow controller. Ensure the flow controller pressure differential, working pressure, flow range, and flow accuracy specifications meet your needs.

— CAUTION —

**Do not use the flow controller valve as an on-and-off valve. Tightening the valve fully clockwise may damage the valve seat.**

• **Flow Diverter**
The flow diverter is a manually operated three-way valve. In the position shown in Figure 2-1, the carrier gas is connected to the instrument; whereas, in the opposite position, the gas is vented to atmosphere via the monitoring port. The flow rate can be checked by connecting a soapbubble flowmeter to the monitoring port.

### 2.4.2 Hydrogen and Air Supplies

The required plumbing for the hydrogen or air supply is shown in Figure 2-2. All of the components are necessary except for the flow diverter. However, the flow diverter does allow the hydrogen or air flow rate to be monitored without disconnecting internal instrument plumbing. The components comprising the hydrogen or air supply are described in the following paragraphs.

• **Gas Supply**
The gas supply is a standard compressed gas cylinder. Use high-purity grade hydrogen and compressed breathing air for the gas supplies.

• **Two-Stage Pressure Regulator**
The two-stage pressure regulator monitors the gas cylinder pressure and regulates the second stage pressure. Set the second stage pressure for 40 to 60 psi.

• **Shut-Off Valve**
The shut-off valve is used to turn the hydrogen or air supply on and off as required. The shut-off

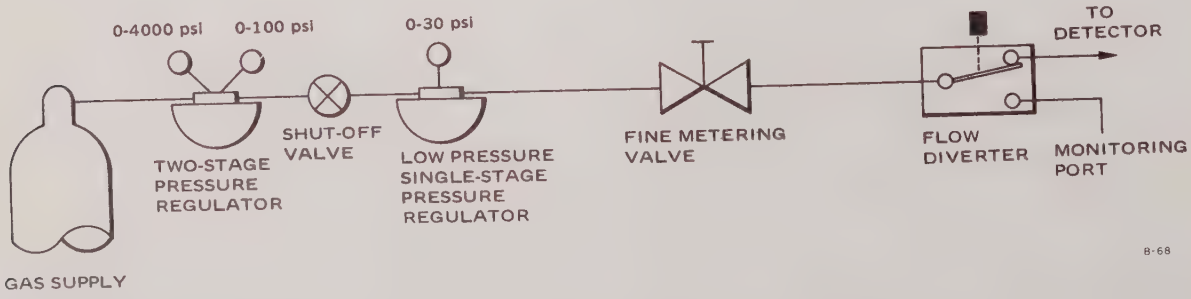



FIGURE 2-2 AFID $H_2$ AND AIR SUPPLIES PLUMBING DIAGRAM

valve eliminates the need for using the fine metering valve as an on-and-off valve which damages the valve seat in the fine metering valve.

• **Single-Stage Pressure Regulator**
The single-stage pressure regulator is used to regulate the gas supply at a low pressure. The two-stage pressure regulator can not properly regulate at second-stage pressures less than 15 psi. Set the single-stage pressure regulator for 5 to 10 psi for hydrogen and 10 to 15 psi for air.

• **Fine Metering Valve**
The fine metering valve is used to set the desired flow rate. Use a high-quality, fine metering valve to obtain optimum flow control.

NOTE

**Do not use the fine metering valve as an on-and-off valve. Tightening the valve fully clockwise may damage the valve seat.**

• **Flow Diverter**
The flow diverter is a manually operated three-way valve. In the position shown in Figure 2-2, the gas is connected to the detector; whereas, in the opposite position, the gas is vented to atmosphere via the monitoring port. The flow rate can be checked by connecting a soapbubble flowmeter to the monitoring port.

## 2.5 INSTALLATION PROCEDURE

Since there are two AFID Kits, there are two installation procedures. Using the appropriate installation procedure, install the AFID as follows:

### 2.5.1 Installation For 1200 and 2100 Series Instruments

1) Turn off the cell voltage at the electrometer.

WARNING

**Ensure Electrometer CELL VOLTAGE switch is turned off and that the cell voltage cable is disconnected from the detector tower to prevent a possible shock hazard.**

2) Turn off power to the detector oven. Allow the detector base and detector tower to cool before proceeding with the installation.

3) Remove the existing detector tower and flame tip.

NOTE

**When installing the AFID in a 1200 Series Instrument, mount the temperature-limit-switch actuator arm to the AFID tower as shown in Figure 2-3. The actuator arm actuates a microswitch which allows the detector oven to heat above 225°C. The 2100 Series Instruments do not require the actuator arm.**

4) Install the salt tip assembly on the detector base.

5) Secure the salt tip assembly to the detector base by tightening the mounting nut fully clockwise.

6) Place the adapter ring flat-side down (recessed side up) on the detector base. Align the holes in the adapter ring with the mounting tabs on the detector base as shown in Figure 2-3.

7) Secure the adapter ring and retaining tabs to the detector base with the mounting screws. Do not tighten the mounting screws at this time.

8) Remove the tower cap from the detector.

9) Set the detector tower onto the adapter ring and detector base. Position the base of the tower inside the adapter ring recess. If the AFID is being installed in a 1200 Series Instrument, ensure that the actuator arm is properly actuating the temperature limit switch.

10) Rotate the retaining tabs clockwise until they engage with the slot in the bottom of the detector tower.

11) Tighten the mounting screws until the detector is firmly held in place.

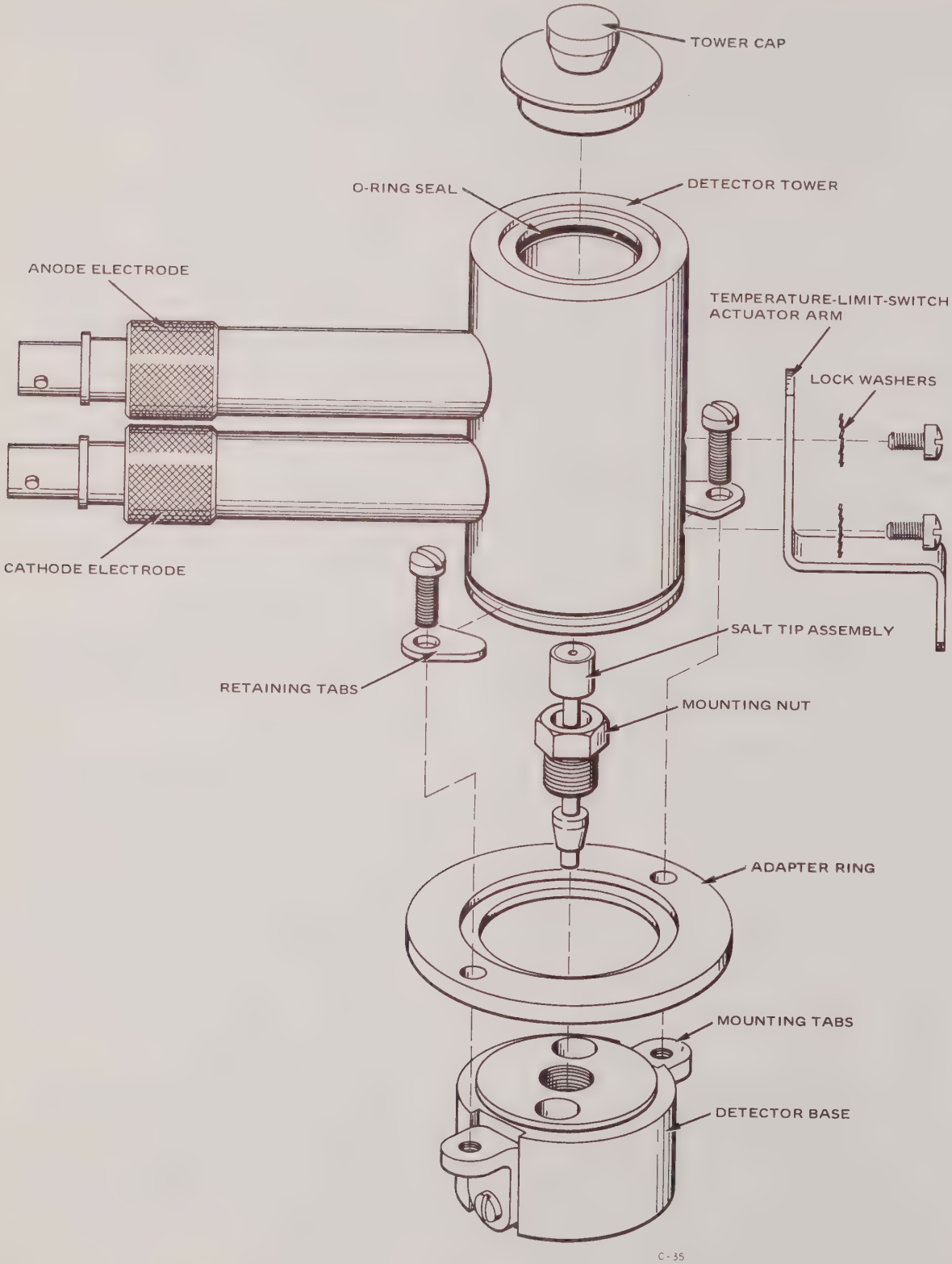



FIGURE 2-3 AFID INSTALLATION ON 1200 and 2100 SERIES INSTRUMENTS

12) Ensure the anode and cathode electrodes are properly positioned with respect to the salt tip assembly as shown in Figure 2-4. The anode electrode position is not that critical, but the cathode electrode position is very critical.

13) Place the cap on the tower and push down to secure in place.

14) Reinstall covers, insulation, lids, and other items removed to gain access to the detector base.

15) Connect the anode (signal) and cathode (cell voltage) cables to the appropriate electrodes. Note that the cell voltage cable has a 2-pin connector and connects to the lower electrode; whereas, the signal cable has a 1-pin connector and connects to the upper electrode.

16) The AFID installation is completed. Refer to Section 4 of this manual for the operating procedure.

**2.5.2 Installation For 1400, 1700, 1800, 2700, and 2800 Series Instruments**

1) Turn off the cell voltage at the electrometer.

> **WARNING**
>
> **Ensure Electrometer CELL VOLTAGE switch is turned off and that the cell voltage cable is disconnected from the detector tower to prevent a possible shock hazard.**

2) Turn off power to the detector oven. Allow the detector base and detector tower to cool before proceeding with the installation.

3) Remove the existing detector tower and flame tip.

4) Install the salt tip assembly on the detector base as shown in Figure 2-5.

5) Secure the salt tip assembly to the detector base by tightening the mounting nut fully clockwise.

6) Remove the tower cap from the detector.

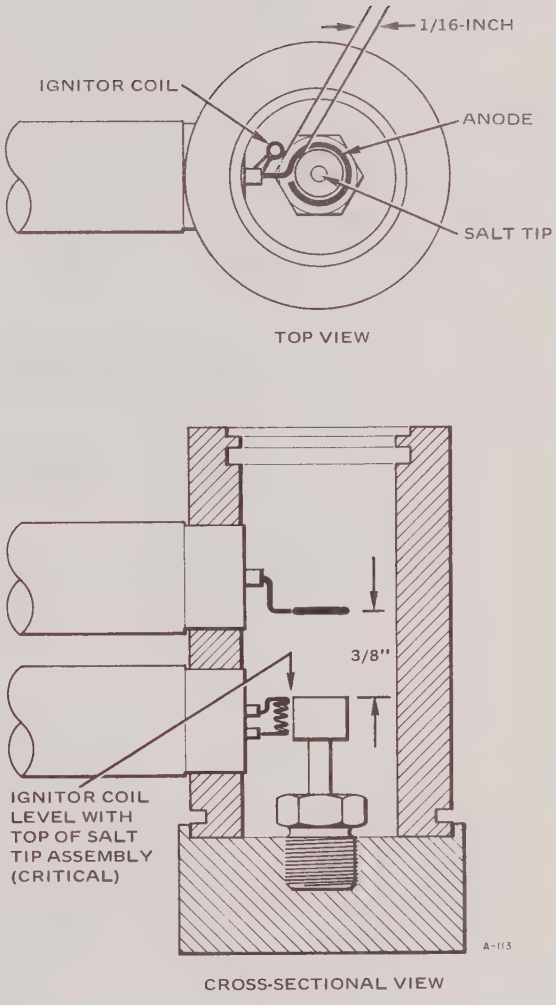



**FIGURE 2-4 AFID ELECTRODE SPACING**

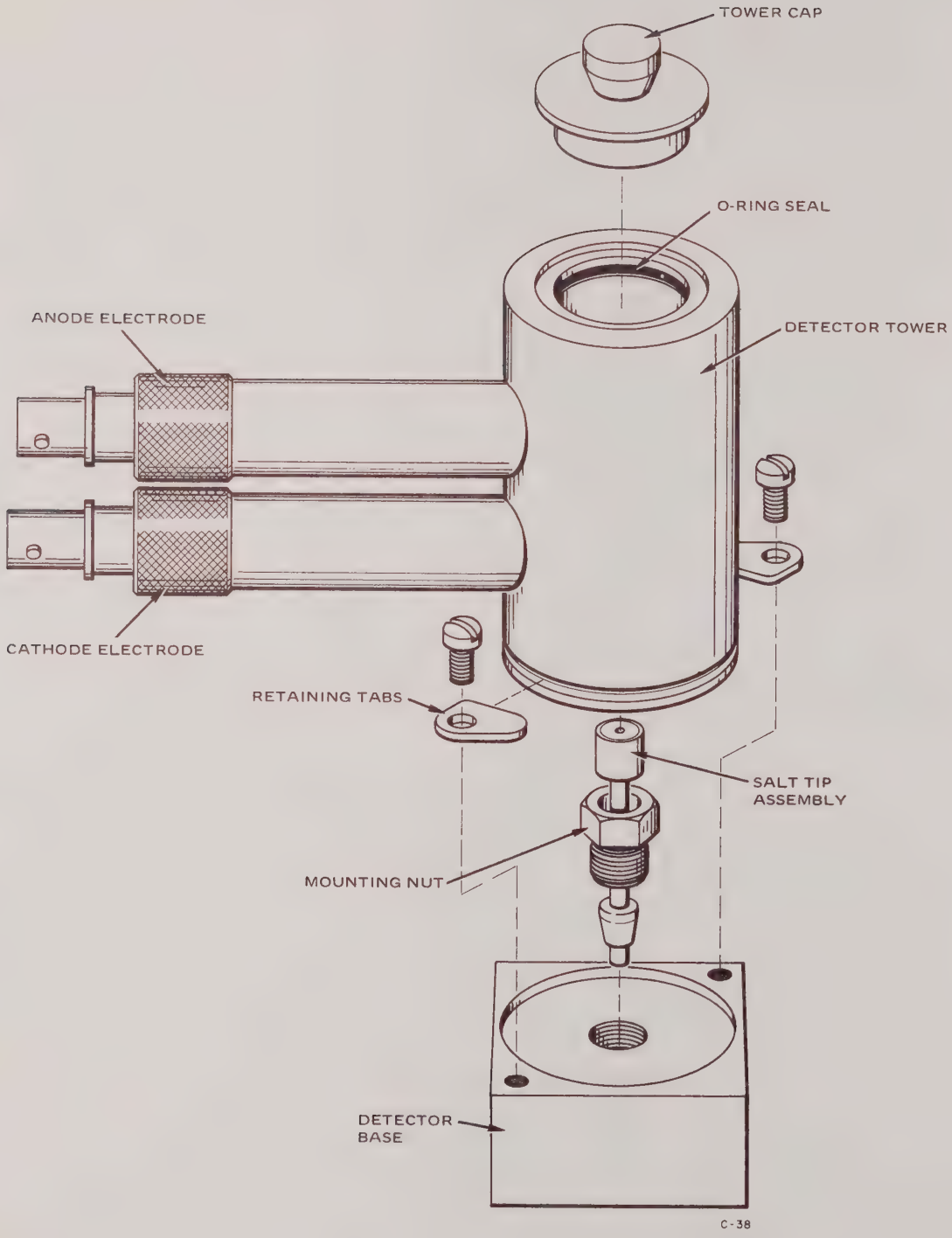



FIGURE 2-5 AFID INSTALLATION ON 1400, 1700, 1800, 2700 and 2800 SERIES INSTRUMENTS

7) Set the detector tower onto the detector base. Position the base of the tower inside the detector base recess.

8) Rotate the retaining tabs clockwise until they engage with the slot in the bottom of the detector tower.

9) Tighten the mounting screws until the detector is firmly held in place.

10) Ensure the anode and cathode electrodes are properly positioned with respect to the salt tip assembly as shown in Figure 2-4. The anode electrode position is not that critical, but the cathode electrode position is very critical.

11) Place the cap on the tower and push down to secure in place.

12) Reinstall covers, insulation, lids, and other items removed to gain access to the detector base.

13) Connect the anode (signal) and cathode (cell voltage) cables to the appropriate electrodes. Note that the cell voltage cable has a 2-pin connector and connects to the lower electrode; whereas, the signal cable has a 1-pin connector and connects to the upper electrode.

14) The AFID installation is completed. Refer to Section 4 of this manual for the operating procedure.

# SECTION 3
# PRINCIPLES OF OPERATION

## 3.1 GENERAL

A precise explanation of the AFID operation is not presently available; however, certain characteristics and operational parameters were obtained during the AFID development. These characteristics, operational parameters, and the detector mechanics are described in the following paragraphs.

## 3.2 BASIC PRINCIPLES OF OPERATION

Although the theory behind the AFID operation is not available, the mechanics of the detector are straightforward. The detector mechanics are as follows: After ignition, a small, cool, hydrogen-rich flame burns at the top of the salt cup. The carrier gas, air, and hydrogen, at the proper flow rates, produce an ionization current (background current) on the order of $3 \times 10^{-11}$ A. When a nitrogen or phosphorus-containing compound passes through the flame plasma, and is ionized, the background current increases proportionally to the concentration of nitrogen or phosphorus. The increase in the background current is sensed by the electrometer through the collector ring above the flame. This signal is amplified to a sufficient level to operate a potentiometer strip-chart recorder.

## 3.3 DETECTOR CONFIGURATION

The detector geometry is an important factor in the operation of the AFID. Therefore, the detector components comprising the AFID, and their function are described in the following paragraphs.

Refer to Figure 3-1 for the following paragraphs.

- **General**

The Alkali Flame Ionization (AFID) construction is similar to the standard Flame Ionization Detector (FID) and therefore, will be likened to the FID.

- **Alkali Salt Tip Assembly**

The standard FID flame tip assembly is replaced with a rubidium sulfate ($Rb_2SO_4$) salt tip assembly. The salt tip assembly provides vaporized $Rb_2SO_4$ necessary for phosphorus and nitrogen detection. Vaporized $Rb_2SO_4$ is produced by burning a hydrogen-air mixture at the center of a bed of $Rb_2SO_4$. The salt tip assembly comprises a metal cup and support tube. Approximately 0.5 g of $Rb_2SO_4$ is pressed into the cup around the support tube. The support tube positions the cup within the detector and provides a passage for the hydrogen, carrier gas, and sample. The support tube also prevents the sample and solvent from directly contacting the $Rb_2SO_4$ which can cause sample loss or peak tailing.

- **Anode Electrode**

The standard FID anode electrode has been modified by replacing the cylinder (collector) with a wire loop. The loop collects the electrons and negative ions produced when a sample is ionized.

- **Cathode Electrode**

The cathode electrode provides an ignitor coil and polarizing voltage. The standard FID cathode electrode has been modified by removing the voltage contact leads which apply the polarizing voltage (-300 V) to the flame tip. The polarizing voltage is not connected to the AFID salt tip assembly because the salt tip assembly is at ground potential. The ignitor coil is sufficiently heated (electrically) to ignite the hydrogen-air mixture. The polarizing voltage minimizes the collection of ions from the vaporized salt while providing optimum collection of the desired sample ions. Since the polarizing voltage is not applied to the salt tip assembly, it is applied to the ignitor coil which is placed approximately 1/16 inch from the salt cup. It is important that the top of the ignitor coil be level with the top of the salt tip assembly. If set too high, excessive background current is collected. If set too low, insufficient background current is collected and the flame may be difficult to ignite.

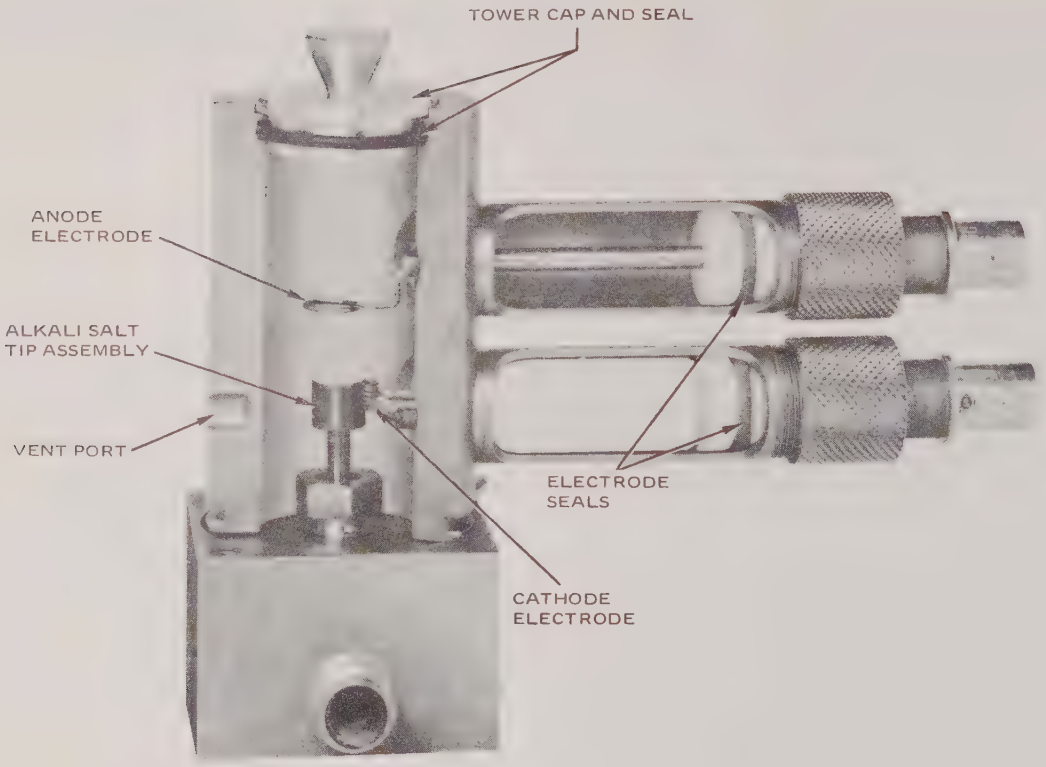



FIGURE 3-1 CROSS-SECTIONAL VIEW OF THE AFID

• **Electrode Seals**
The electrode seals prevent exhausting of gases at the electrodes. This prevents the formation of condensates which can short the electrodes to ground.

• **Tower Cap and Seal**
The standard FID tower and tower cap have been modified to incorporate an O-ring seal. The O-ring seal ensures an air-tight seal between the tower and tower cap. The purpose of this seal is to divert the flame-support air from its normal path to a short circuit path on the side of the tower. The seal also prevents the back diffusion of ambient air into the cell chamber. Rerouting the flame-support air results in the air reaching the flame by diffusional flow rather than convection flow; thereby, minimizing excessive background current and noise.

• **Vent Port**
The vent port has been precisely located in the tower wall to provide sufficient purging of the detector chamber gases and to provide the new flame-support air flow path.

## 3.4 OPERATIONAL PARAMETERS

Without a precise theory of operation available, a complete understanding of the AFID operational parameters is necessary to properly operate the detector. The following operational parameters were determined during the development of the detector.

### 3.4.1 General

The AFID operation is dependent on the ionization of rubidium sulfate ($Rb_2SO_4$). The ionization current (background current) is an indication of the detector sensitivity. Generally, with ionization detectors, the higher the background current the greater the detector sensitivity. However, the AFID noise level increases disproportionately with an increasing background current. Therefore, the background current maximum and signal-to-noise ratio are set for optimum detector operation rather than just maximum. Since the background current is a function of the hydrogen-air mixture, the hydrogen and air supplies require special considera-

tion. The following paragraphs cover the hydrogen and air supplies and other detector parameters to be considered before operating the detector.

NOTE

**It is desirable to provide a means of measuring the hydrogen, air, and carrier gas flow rates without disassembling the detector (such as flow diverters).**

### 3.4.2 Hydrogen Supply

The hydrogen supply provides the necessary combustible gas for the flame. The optimum hydrogen flow rate may vary daily depending on the operating conditions and the type of analysis. The hydrogen flow rate should be more precisely controlled than the air flow rate. The detector background current responds to as little as 0.01 ml/min change in the hydrogen flow rate (at maximum sensitivity). Since the detector is sensitive to changes in the hydrogen flow rate, the hydrogen supply must be regulated. Usually, a good quality, two-stage pressure regulator and needle valve will suffice to control the hydrogen from a compressed gas cylinder. However, ensure that only metal tubing is used in the plumbing between the detector and gas cylinder. Since the needle valve must have precise control of the hydrogen flow rate, the valve head pressure should be 5 to 10 psi. The lower the valve head pressure, the more control the valve has on the hydrogen flow rate; however, the second stage of the regulator must be able to regulate within this pressure range.

As mentioned before, the optimum hydrogen flow rate for one detector will not necessarily be optimum for another detector. Each AFID must be calibrated independently by optimizing the hydrogen and air flow rates for the best signal-to-noise ratio (sensitivity).

### 3.4.3 Air Supply

The air flow serves the dual function of supporting combustion of the hydrogen flame and purging the detector of burned products. The optimum air flow rate may vary as operating conditions and analyses are changed. Since the detector is sensitive to air flow rate changes (0.1 ml/min at maximum sensitivity), the air supply system must be regulated. Usually, a good quality, two-stage pressure regulator and needle valve, in an all-metal plumbing system, will suffice to control the air from a compressed gas cylinder. To obtain precise control of the air flow rate with the needle valve, the valve head pressure should be 10 to 15 psi. Ensure that the second stage of the regulator will regulate within the desired pressure range.

### 3.4.4 Carrier Gas Supply

The recommended carrier gas flow rate range is 15 to 40 ml/min for 1/8-in columns and 40 to 60 ml/min for 1/4-in columns. Slightly higher carrier gas flow rates have been used successfully; although, the low end of the range is preferred. Since the detector is sensitive to changes in the carrier gas flow rate, the carrier gas supply must be regulated. Usually, a good quality, two-stage pressure regulator and needle valve, in an all-metal plumbing system, will suffice to control the carrier gas from a compressed gas cylinder (needle valve and/or flow controller is provided on most VA instruments). Both helium and nitrogen can be used for carrier gas; however, helium is preferred.

### 3.4.5 Detector Temperature

The maximum operating temperature used during the development of the AFID was 280°C. Operating at higher temperatures requires caution since the affects on the $Rb_2SO_4$ pellet are unknown at this time. Temperature changes caused by room drafts and air conditioning ducts may result in random baseline fluctuations and detector instability. To reduce temperature change effects, cover the portion of the detector outside of the detector oven with a piece of insulation.

# SECTION 4
# OPERATION

## 4.1 GENERAL

The purpose of the OPERATION section is to familiarize the operator with the function and operation of the AFID detector. Before operating the detector, read the complete OPERATION section to become familiar with the AFID terminology and characteristics.

## 4.2 DETECTOR CHARACTERISTICS

The following information will familiarize the operator with the AFID characteristics. Understanding the detector characteristics will assist the operator in selecting the proper operational parameters.

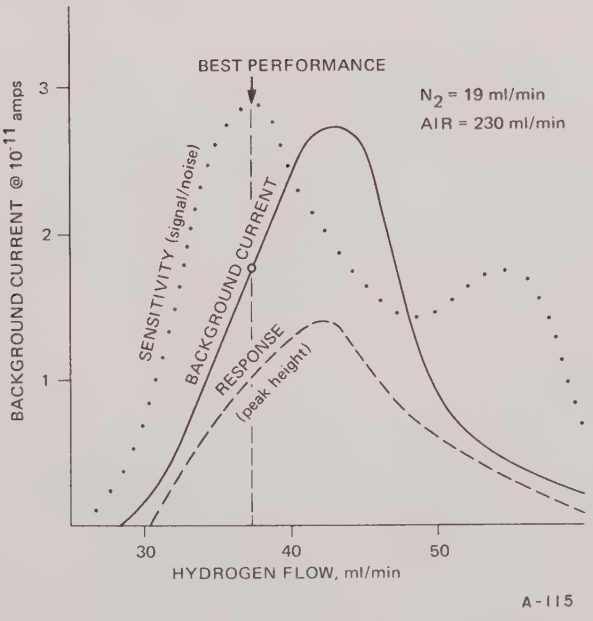



FIGURE 4-1 AFID CHARACTERISTICS VERSUS $H_2$ FLOW RATE

**• AFID Characteristics versus $H_2$ Flow Rate**

The curves in Figure 4-1 illustrate the relationship between background, response and sensitivity with the air flow optimized at 230 ml/min. The background current curve and response curve (coulomb/gram) are maximum at a hydrogen flow rate of 42 ml/min. However, the background current and response curves, by themselves, do not indicate the optimum point because the noise level has not been considered. The detector sensitivity (signal-to-noise) is the important detector characteristic which must be optimized. The sensitivity curve shown in Figure 4-1 was obtained by dividing the response by the noise level at each of the hydrogen flow rates. For a wide variety of nitrogen and phosphorus-containing organics, the best sensitivity occurs at a hydrogen flow that yields a background current of approximately half the maximum obtainable. Also, the solvent response exhibits much better recovery at lower hydrogen flow rates.

**• Linearity**

As shown in Figure 4-2, a linear response was obtained from a sample size range of 0.15 ng to 150 ng of Azobenzene.

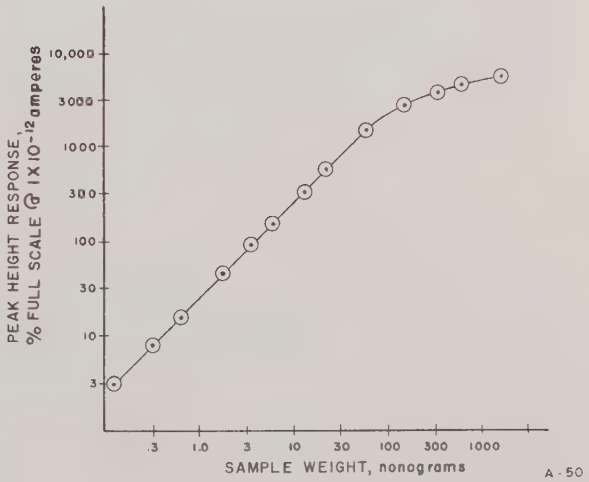



FIGURE 4-2 AFID LINEARITY FOR AZOBENZENE

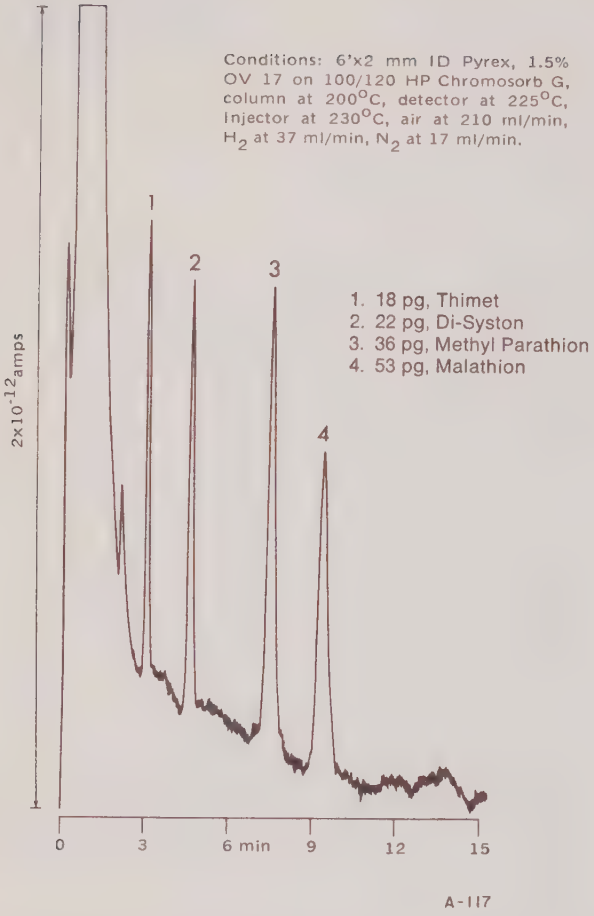

Conditions: 6'x2 mm ID Pyrex, 1.5% OV 17 on 100/120 HP Chromosorb G, column at 200°C, detector at 225°C, injector at 230°C, air at 210 ml/min, $H_2$ at 37 ml/min, $N_2$ at 17 ml/min.
1. 18 pg, Thimet
2. 22 pg, Di-Syston
3. 36 pg, Methyl Parathion
4. 53 pg, Malathion
$2 \times 10^{-12}$ amps
0
3
6 min
9
12
15
A-117

FIGURE 4-3 PICOGRAM DETECTION WITH THE AFID

• **Sensitivity**
The AFID sensitivity is 10 X $10^{-13}$ g/sec for nitrogen-containing compounds; 0.15 X $10^{-13}$ g/sec for phosphorus-containing compounds; and 100,000 X $10^{-13}$ g/sec for normal hydrocarbons. The AFID sensitivity for specific compounds is illustrated in Figure 4-3 and Table 4-1.

• **Specificity**
The selectivity of response of the AFID is variable, depending on the hydrogen flow. Generally, it has a discrimination factor of 10,000 to 1 against organics other than organophosphates. However, larger factors, 50,000 to 1, can be obtained by fine tuning the hydrogen flow. Also, the AFID is 10,000 times less sensitive to normal hydrocarbons than the standard Flame Ionization Detector (FID). Examples of the AFID and FID responses to tertiary amines, extracted from three different blood plasma samples, are shown in Figure 4-4. The molecular structure of the extracted compounds are shown in Figure 4-5.

• **Background Current**
The AFID background current is 3 X $10^{-11}$ A.

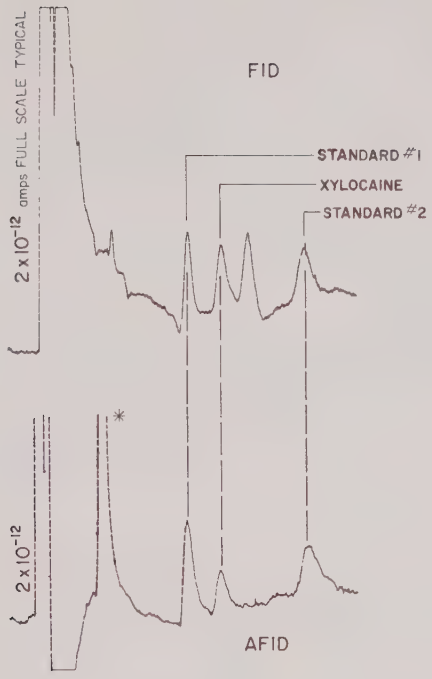
• **Noise Level**
The AFID noise level is 1 to 3% of full scale at 1 X $10^{-12}$ A when the anode and cathode electrodes are properly positioned.

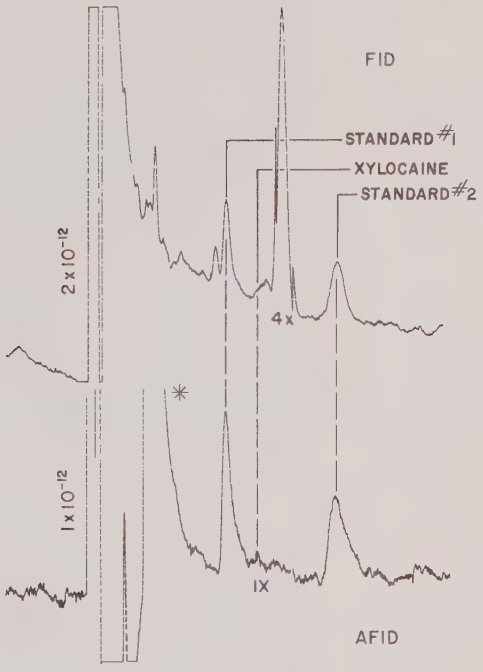
• **Stability**
One of the unique advantages of the Varian Aerograph AFID is that once the detector has equilibrated, the response does not appreciably change with time. In fact, the standard procedure is to allow the AFID flame to burn continuously, day after day, as with the FID. The controlled rate of evaporation of the salt is slow and the response is not affected.

• **Solvent Response**
Some of the recommended solvents for the AFID, are: methyl acetate, ethyl acetate, and ethyl ether. Also, water and methanol may be used; however, they are not compatible with silanized column supports. The water yields a slight negative response with rapid recovery; whereas, methanol and ethyl ether yield a positive response followed by a nega-

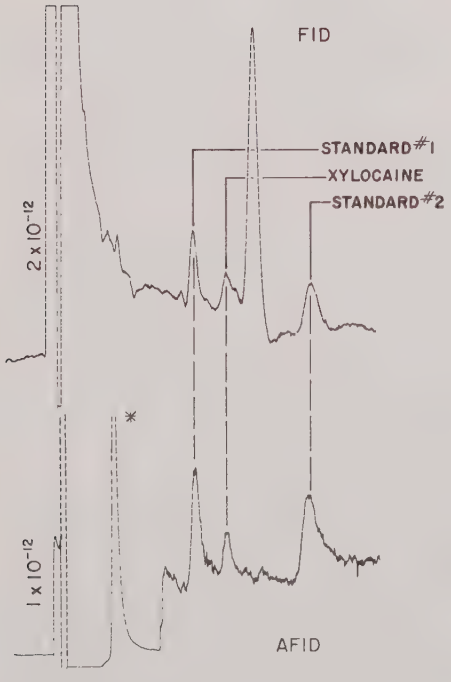
TABLE 4-1
AFID SENSITIVITY

| Sample | Minimum Detection (pg) | Pesticide Molecule (g/sec) | Atomic Phosphorus (g-atom/sec) |
|---|---|---|---|
| Thimet | 0.4 | 2.8 X $10^{-14}$ | 3.3 X 10-15 |
| Di-Syston | 0.6 | 3.3 X $10^{-14}$ | 3.8 X $10^{-15}$ |
| Ronnel | 0.9 | 3.3 X $10^{-14}$ | 3.2 X $10^{-15}$ |
| Methyl Parathion | 0.9 | 3.1 X $10^{-14}$ | 3.7 X $10^{-15}$ |
| Parathion | 1.3 | 3.7 X $10^{-14}$ | 4.2 X $10^{-15}$ |
| Malathion | 2.0 | 5.2 X $10^{-14}$ | 5.4 X $10^{-15}$ |




*TRIBULYL PHOSPHATE INADVERTENTLY ADDED WHILE PREPARING SAMPLE.




## CHROMATOGRAPHIC CONDITIONS

- **Instrument**
  Varian Aerograph Model 2100.

- **Column**
  5' X ¼" Pyrex (2mm ID), 3% OV 17 on high performance Chrom W 100/120 mesh, silanized with three 10μl injections of Silyl 8 (column effluent to atmosphere) before analysis.

- **Temperatures**
  225°C injector
  200°C column
  225°C detector

- **Flows**
  19 ml/min helium
  43 ml/min hydrogen
  265 ml/min air

- **Sample Size**
  2 μl

FIGURE 4-4 AFID SELECTIVITY

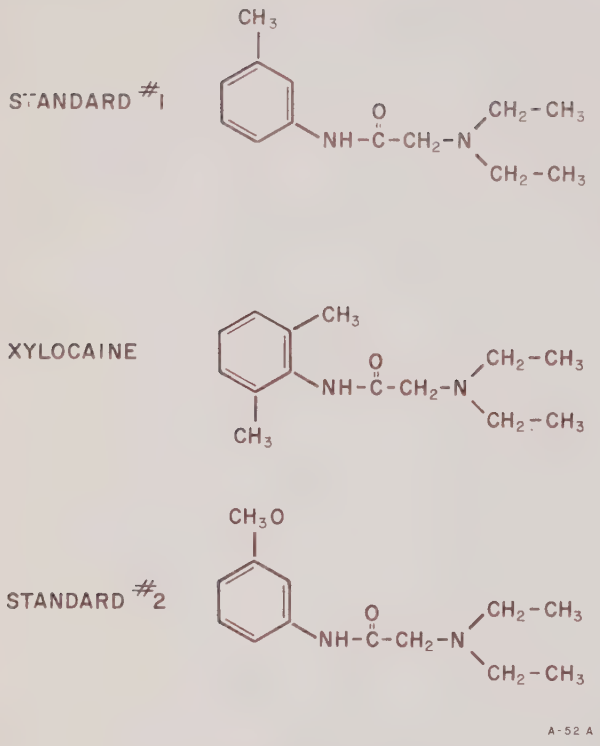



FIGURE 4-5 MOLECULAR STRUCTURE OF COMPOUNDS STUDIED

tive recovery response (2 min for a 2 µl injection). The hydrocarbon solvents, e.g., hexane, iso-octane, and petroleum ether, will extinguish the flame when more than 2 µl are injected, but are otherwise acceptable. Solvents that are completely unsatisfactory because of their excessive recovery time are: carbon disulfide, carbon tetrachloride, and chloroform.

A very interesting characteristic of the AFID is the solvent response at different hydrogen flow rates. As illustrated in Figure 4-6, six injections of 100 ng of Xylocaine* dissolved in 2 µl of methanol were made at different hydrogen flow rates. At hydrogen flow rates above 50 ml/min, the methanol tailed into the Xylocaine; whereas, at flow rates below 50 ml/min, the methanol response is fully recovered before the Xylocaine begins to elute. The hydrogen flow rate at 44 ml/min is optimum since the best signal-to-noise ratio (noise level not shown) and minimal response were obtained at this flow rate.

*U.S. Patent No. 2,441,001 assigned to ASTRA.

## 4.3 ROUTINE OPERATION

Operate the AFID as follows:

1) Ensure the carrier gas, hydrogen, and air supplies are connected to the instrument as described in Section 2.

2) Set the hydrogen flow rate for 35 ml/min ± 2 ml/min.

3) Set the carrier gas for the desired flow rate. The type of analysis to be made will determine the desired flow rate.

4) Set the air flow rate for 235 ml/min ± 10 ml/min.

5) Install the AFID as described in Section 2.

6) Ensure the AFID electrodes are positioned as described in Section 2.

7) Set up the required injector, detector, and column-oven temperatures.

8) Shunt the recorder input. Adjust recorder pen for mechanical and electrical zero.

9) Balance the electrometer as described in the appropriate instruction manual.

10) Set electrometer CELL VOLTAGE control to the flame position (FL).

11) Set electrometer RANGE control to F1 X $10^{-12}$ position (1 X $10^{-12}$ A full scale).

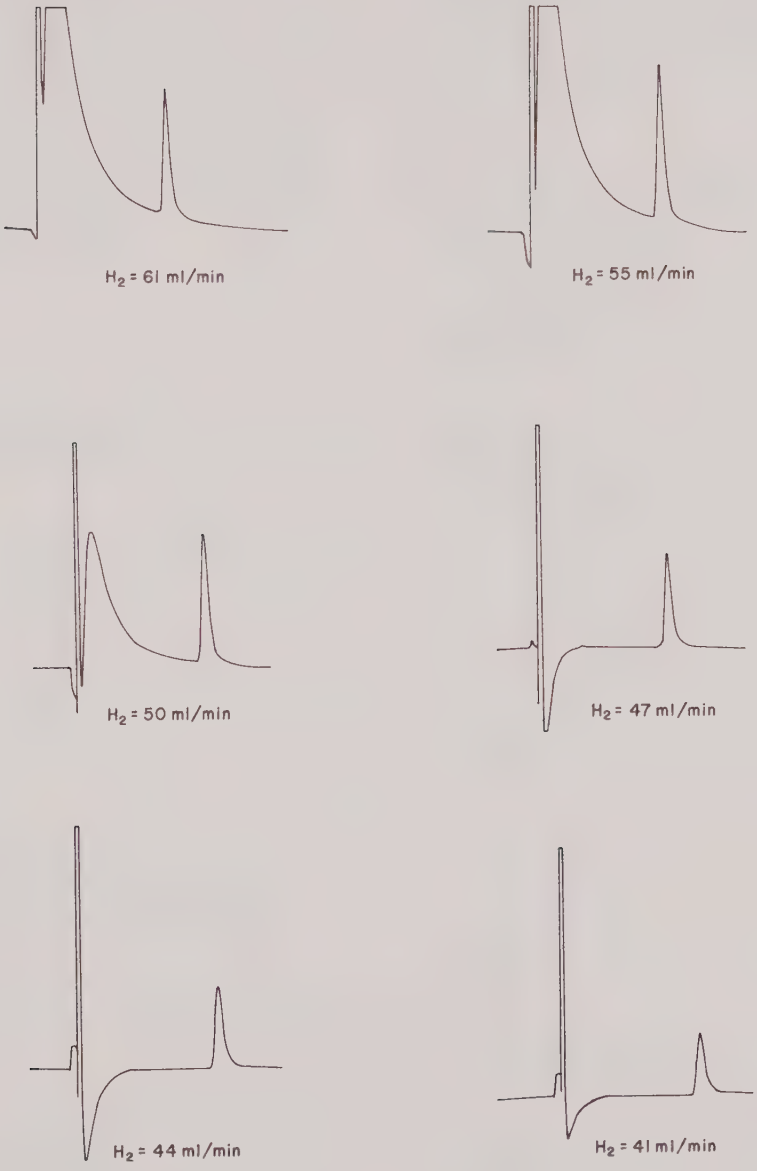
12) Set electrometer ATTENUATOR control to the 32 position (32 X $10^{-12}$ A full scale).

13) Set electrometer MODE control to the appropriate flame position:

FL: Single Channel Electrometer
FL-FL: Dual Channel Electrometer

**WARNING**

**A potential operator hazard exists if the detector cap is not removed prior to igniting the hydrogen-air mixture. The detector cap will be blown off when the flame is ignited. Therefore, always remove the cap prior to igniting the flame.**




NOTE: Chromatograms Run At $32 \times 10^{-12}$ Amps Full scale

A-64

FIGURE 4-6 AFID SOLVENT RESPONSE AS A FUNCTION OF HYDROGEN FLOW

14) Remove detector-tower cap.

15) Ignite the flame and immediately (press cap down slowly, otherwise flame will blow out from air pressure) replace the detector cap. Excessive air results in a hot flame that can damage the salt pellet.

— CAUTION —

**Do not allow the background current to exceed 128 X $10^{-12}$ A longer than 3 minutes. Excessive background current will damage the salt pellet. If necessary, decrease the air flow rate to reduce the background current.**

16) After igniting the flame, allow 30 min for the background current to stabilize. It may be necessary to momentarily increase the attenuation to bring the baseline onto scale.

NOTE

**After igniting the flame, the recorder pen will drift up scale. This indicates the flame is lit and the salt pellet is heating. If the recorder pen does not drift up scale, increase the air flow rate. If the recorder pen does not respond to the increase in air, the flame is not lit.**

17) Optimize the hydrogen-air mixture as follows:

a) Adjust the air flow rate for a background current near the baseline at 32 X $10^{-12}$A.

b) Increase the hydrogen flow rate in small steps as shown in Figure 4-7. Allow the background current to equilibrate after each flow rate adjustment.

c) As long as the recorder pen moves up scale, continue increasing the hydrogen flow rate. To keep the recorder pen on scale, reduce the air flow rate as required.

d) When the recorder pen begins to move down scale, stop increasing the hydrogen flow rate. Reduce the hydrogen flow rate slightly to obtain the maximum up scale deflection.

e) If the background current (obtained in Step 17-d) is not 80 to 100% of full scale at 32 X $10^{-12}$ A, increase the air flow rate for maximum background current (do not exceed 32 X $10^{-12}$ A). If the background current is still low, increase the hydrogen flow rate to obtain the proper amount of background current.

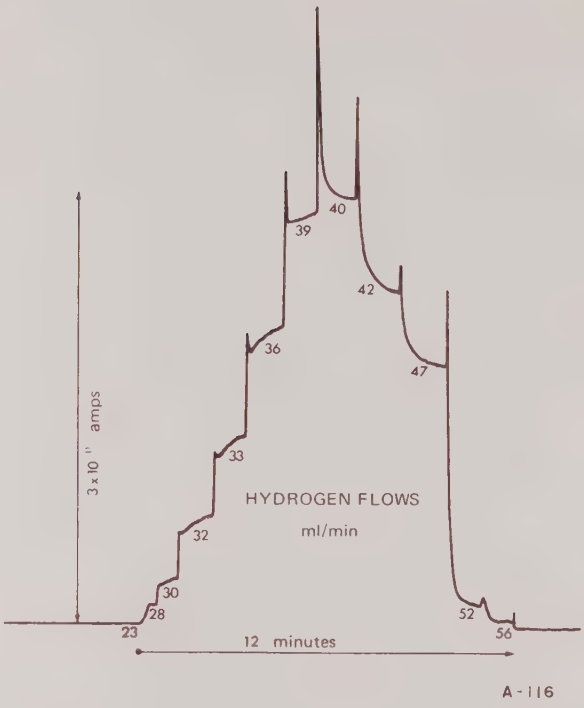



FIGURE 4-7 HYDROGEN OPTIMIZATION OF AFID

18) It may be necessary to repeat Step (17) several times to properly optimize the hydrogen-air mixture.

19) After obtaining the proper background current, decrease the hydrogen flow rate until the background current is reduced to ≈50% of the maximum established in Step (17).

20) Allow ≈5 min for the baseline to stabilize.

21) Buck out the background current with the appropriate electrometer BUCKING control.

22) The AFID is now ready to operate. Slight adjustments of the air and hydrogen flow rates may be required to maintain optimum AFID operation.

— CAUTION —

**Silicon from TMS derivatives or poorly conditioned silicon-polymer columns are converted to $SiO_2$ in the flame. The $SiO_2$ coats the salt tip which causes the background current to slowly decrease. As the background current decreases, the detector sensitivity also decreases. To restore detector background current and sensitivity, lightly burnish the slat tip to remove the $SiO_2$ coating.**

## 4.4 OPERATIONAL HINTS

The following paragraphs describe special operating techniques developed during the AFID design.

**• Hydrogen and Air Flows**
Ensure that the hydrogen and air supplies have high-quality pressure regulators and needle valves in an all-metal plumbing system.

When instruments are not equipped with flow diverters, make note of valve settings so you can return to original settings without disassembling the detector to check flow rates.

**• Background Current**
The following are possible causes of negative background current:

a) Anode electrode too far from the salt tip.

b) Ignitor coil higher than the salt tip.

c) Contaminated chromatography system.

**• Response**
The AFID yields a positive response to phosphorus or nitrogen-containing compounds; however, a negative response may occur when other molecular combinations of functional groups (e.g., sulfur and chlorine) are present in the sample.

**• Operation With Series 1200 Instruments**
The detector-base heater, in Series 1200 Instruments, is controlled by a bi-metalic thermostat. As the thermostat cycles, the detector-base temperature cycles, and the temperature of the hydrogen line in the base cycles; thereby causing the hydrogen flow rate to cycle. This cycling of the hydrogen flow rate does not affect the standard FID, but may cause the AFID baseline to cycle $\approx$20% at 32 X $10^{-12}$ A. To minimize the baseline cycling to an acceptable level, replace the existing detector-base heater (150W) with a 100W cartridge heater.

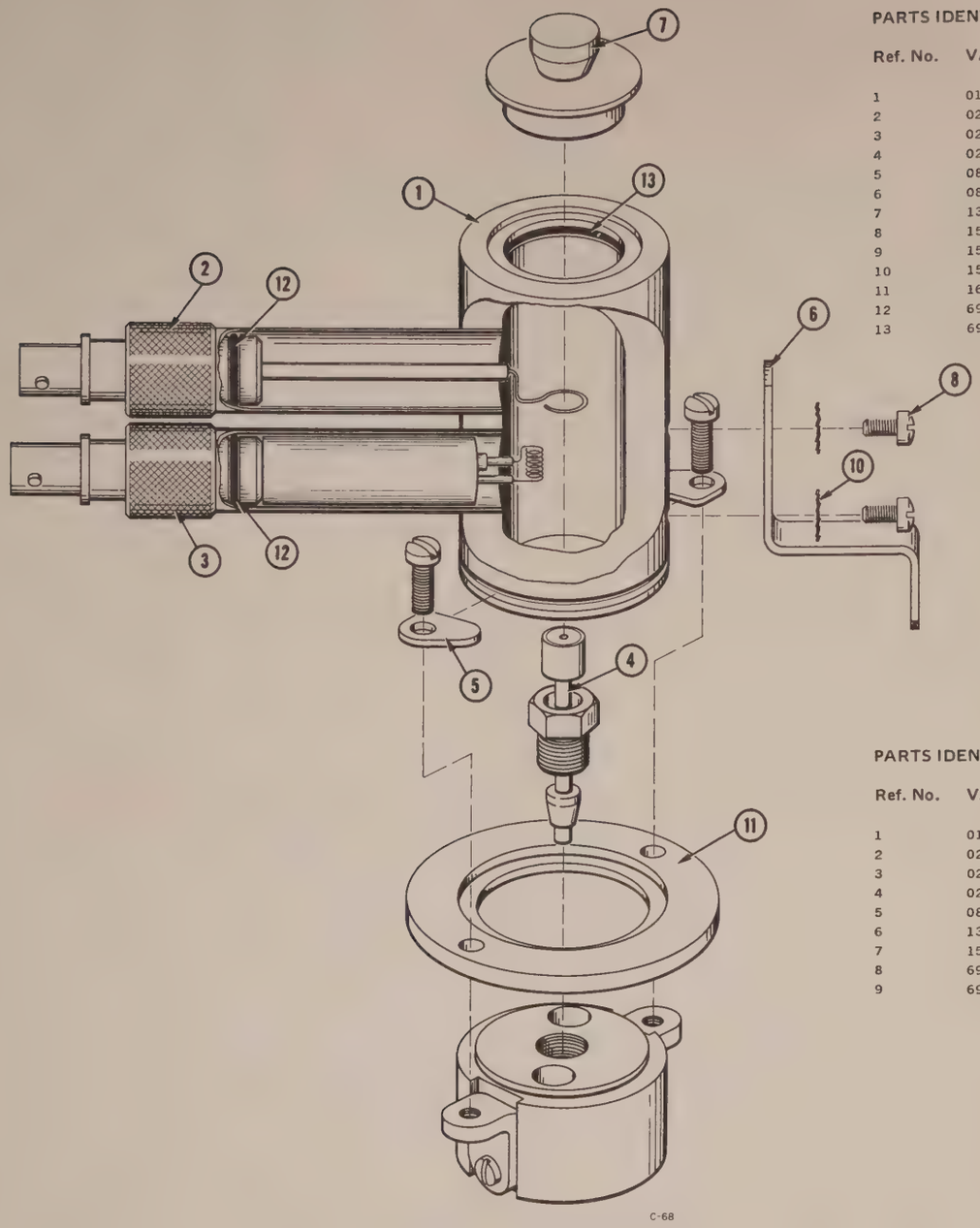
# SECTION 5
# ILLUSTRATED PARTS LISTS

## 5.1 GENERAL

This section of the manual comprises the AFID Kit Assembly Drawings and Parts List. The parts are identified on the assembly drawings by a reference number within a circle. The reference number is listed numerically in the parts list. The number is followed by a corresponding part number and material description. Use the ten-digit part number and material description when ordering replacement parts.

Listed below are the titles, drawing numbers, and page numbers of the assembly drawings in this manual.


| TITLE | DWG. NO. | PAGE NO. |
|---|---|---|
| AFID Kit for 1400, 1700, 1800, 2700 and 2800 Series Instruments | 02-001351-00 | 5-2 |
| AFID Kit for 1200 and 2100 Series Instruments | 02-001352-00 | 5-2 |



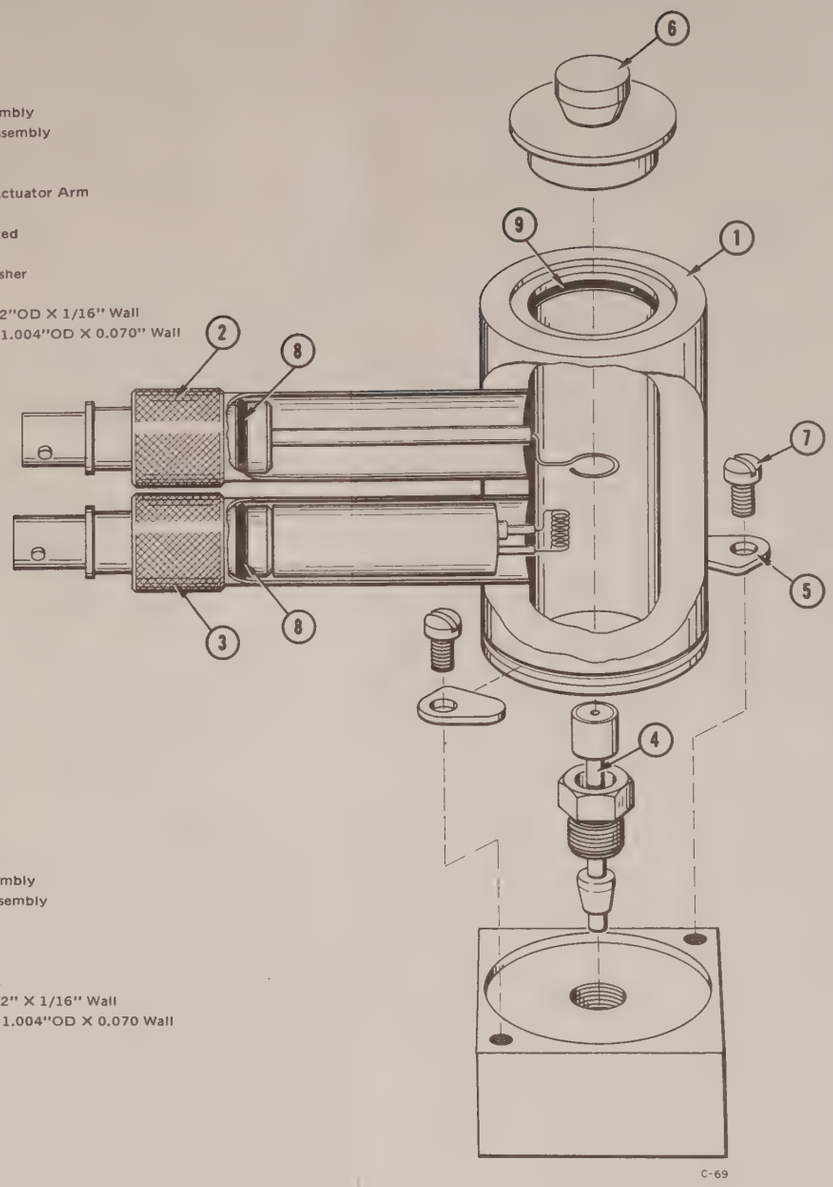

AFID KIT FOR 1200 AND 2100 SERIES INSTRUMENTS 02-001352-00

PARTS IDENTIFICATION FOR 02-001352-00

| Ref. No. | VA Part No. | Description |
|---|---|---|
| 1 | 01-000791-00 | AFID Tower Assembly |
| 2 | 02-001363-00 | AFID Anode Electrode Assembly |
| 3 | 02-001364-00 | AFID Cathode Electrode Assembly |
| 4 | 02-001365-00 | AFID Salt Tip Assembly |
| 5 | 08-000072-00 | Tower Retaining Tab |
| 6 | 08-000162-00 | Temperature-Limit-Switch Actuator Arm |
| 7 | 13-000048-00 | AFID Tower Cap |
| 8 | 15-000128-00 | 6-32 X 1/4" BHMS, Cad Plated |
| 9 | 15-000165-00 | 8-32 X 3/8" PHMS, Stn. Stl. |
| 10 | 15-003261-00 | #6 External Tooth Lock Washer |
| 11 | 16-000624-00 | Tower Adapter Ring |
| 12 | 69-000025-00 | Silicone O-ring, 3/8"ID X 1/2"OD X 1/16" Wall |
| 13 | 69-000102-00 | Silicone O-ring, 0.864"ID X 1.004"OD X 0.070" Wall |

PARTS IDENTIFICATION FOR 02-001351-00

| Ref. No. | VA Part No. | Description |
|---|---|---|
| 1 | 01-000791-00 | AFID Tower Assembly |
| 2 | 02-001363-00 | AFID Anode Electrode Assembly |
| 3 | 02-001364-00 | AFID Cathode Electrode Assembly |
| 4 | 02-001365-00 | AFID Salt Tip Assembly |
| 5 | 08-000072-00 | Tower Retaining Tab |
| 6 | 13-000048-00 | AFID Tower Cap |
| 7 | 15-004722-00 | 8-32 X 1/4" FHMS, Stn. Stl. |
| 8 | 69-000025-00 | Silicone O-ring, 3/8"ID X 1/2" X 1/16" Wall |
| 9 | 69-000102-00 | Silicone O-ring, 0.864"ID X 1.004"OD X 0.070 Wall |




AFID KIT FOR 1400, 1700, 1800, 2700
AND 2800 SERIES INSTRUMENTS
02-001351-00

varian aerograph

2700 mitchell drive/walnut creek/california 94598/415-939-2400
specialists in chromatography and data handling systems

# FLAME IONIZATION DETECTOR

# INSTRUCTION MANUAL

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Manufactured in the United States of America

PUBLICATION NO. 85-001118-12

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# SECTION 1
# INTRODUCTION

## 1.1 GENERAL

The Varian Aerograph™ flame ionization detector (FID) described in this manual is designed for use in the 2800, 2700, and 1400 Series Gas Chromatographs manufactured by Varian Aerograph. The FID responds to all organic compounds but does not respond to the fixed gases or water. The detector offers excellent operating stability at temperatures up to 400°C. Refer to Table 1-1 for FID specifications.

TABLE 1-1 FID SPECIFICATIONS

**Detectability:**
$9 \times 10^{-13}$ gm/sec: a measure of the smallest detectable quantity.

**Sensitivity:**
.01 coulombs/gm; a measure of the detector's ionization and ion collection efficiency using air to support combustion.

**Linear Range:**
$3 \times 10^{7}$

**Carrier Gas:**
$N_2$, He, or Ar.

**Temperature Limit:**
400°C.

# SECTION 2
# INSTALLATION

## 2.1 GENERAL

The following paragraphs describe installation procedures for the FID. There is one detector base in the 1400 Series and two bases in the 2740, 2840, and 2860 Series instruments. The installation procedures are essentially the same regardless of whether your instrument has one or two detector bases.

**• Installation**

Before installing the detector, make sure the Electrometer CELL VOLTAGE switch is turned OFF and that any previously installed detector is removed; then, proceed as follows:

1) On *2840 and 2860 Series instruments,* remove snap-on detector oven lid, insulation pad, and oven cover plate on the 2800 Series instruments.

   On *1400 Series instruments,* lift up column-oven door.

2) Once installed, the flame tip, ferrule and mounting nut comprise the permanent flame tip assembly. Refer to Figures 2-1 and 2-2 to assemble, if necessary. Install flame tip assembly in detector base. Tighten mounting nut approximately 3/4 turn past finger tightness.

3) Identify detector tower. Loosen knurled nut on cathode probe housing, then retract cathode probe about one inch. Loosen screws securing two tabs to detector base. Rotate tabs to side so bottom of tower will seat properly on detector base. Orient tower so probes point toward rear of instrument on 2840 and 2860 Series instruments and toward side of instrument on 1400 Series instruments. Place tower on detector base, then rotate tabs into slot cut around lower part of detector tower. Tighten tabs with screws to secure tower. Slowly slip cathode probe back into housing making sure prongs on end of probe slip over flame tip. Make sure probe is oriented as shown in Figure 2-1.

4) Identify collector cylinder from Figure 2-1. Slip short end (referenced to slot cut in cylinder) of cylinder down into tower and between clips of collector probe. Make sure clips of collector probe slip into slot cut around collector cylinder and that cylinder is aligned vertically and centered over flame tip. Use a pair of long-nose pliers when removing or installing collector cylinder.

   NOTE

   **The collector cylinder must be removed before the collector probe can be removed.**

5) Install second detector (2840 and 2860 Series only), if desired, at this time.

6) On *2840 and 2860 Series instruments*, replace detector oven cover plate, insulation pad, and oven lid. Place detector cover over detector tower. Make sure tower cap (part of cover) slips into top part of tower. Connect signal and cell voltage cables to appropriate probe, i.e., 1-pin connector (part of signal cable) to collector probe and 2-pin connector (part of cell voltage cable) to cathode probe. These cables are located at the rear of the 2840 and 2860 Series instruments.

   *On 1400 Series instruments,* place cap on detector tower and connect signal and cell voltage cables to appropriate probe, i.e., 1-pin connector (part of signal cable) to anode probe and 2-pin connector (part of cell voltage cable) to cathode probe. These cables can be found just behind the accessory panel near where the detector is installed. Removing right side of cabinet will facilitate connecting or disconnecting of the detector cables.

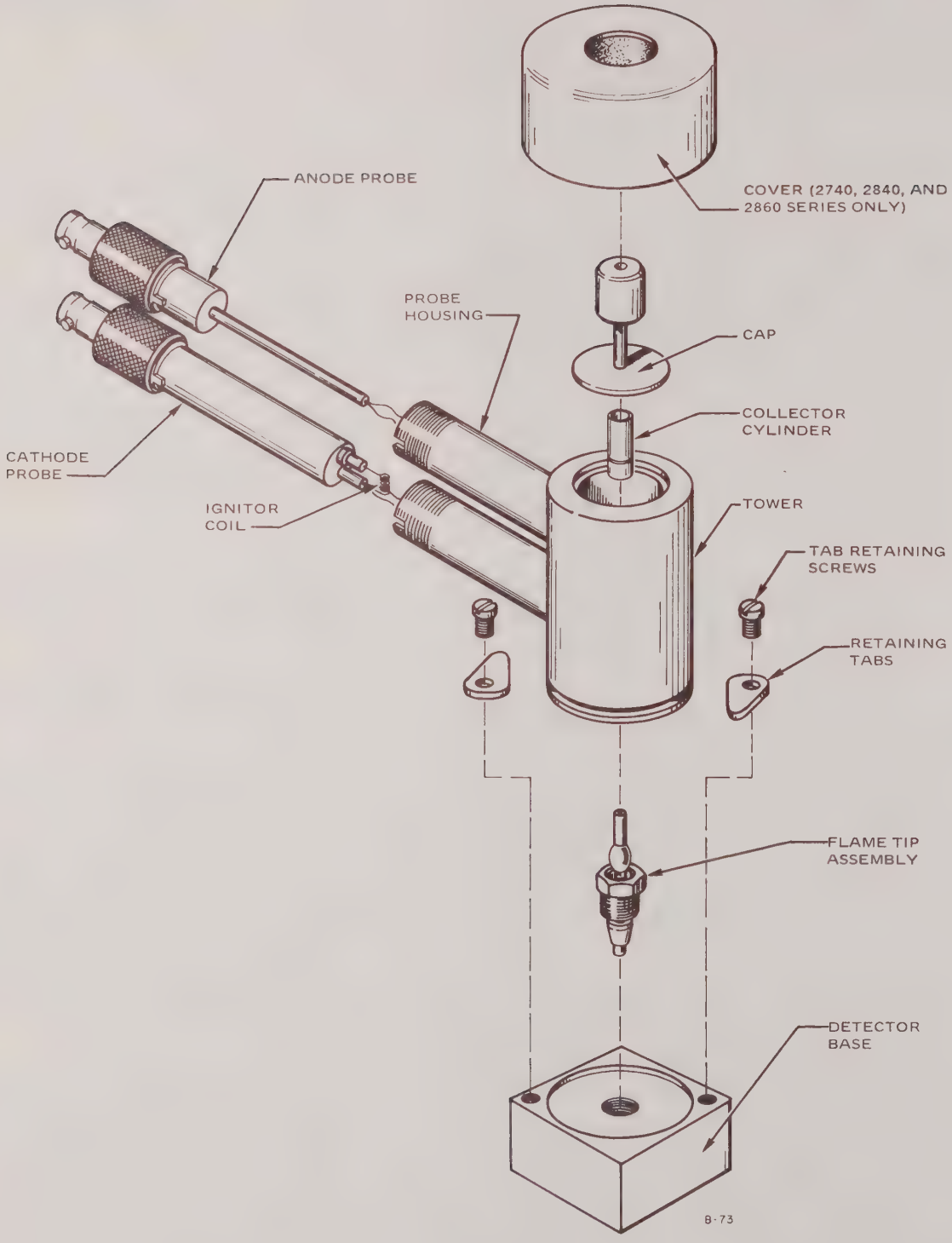



FIGURE 2-1 FID DETECTOR INSTALLATION

Remove right side panel as follows: Grasp top of side panel, then pull panel toward yourself about 1/2 inch. This will loosen panel from guide pins. Now slide panel toward rear of instrument until side panel slips off bottom guides. Remove panel now by pulling away from instrument. Installation of panel is just the reverse of removal.

**• Removal**

Removal of flame detector is just the reverse of the installation procedures. Be very careful when handling the flame tip assembly as this component is very fragile. Complete removal of the probes is also not recommended unless they are to be cleaned or replaced. Be careful not to bend probes when removing or installing in probe housings.

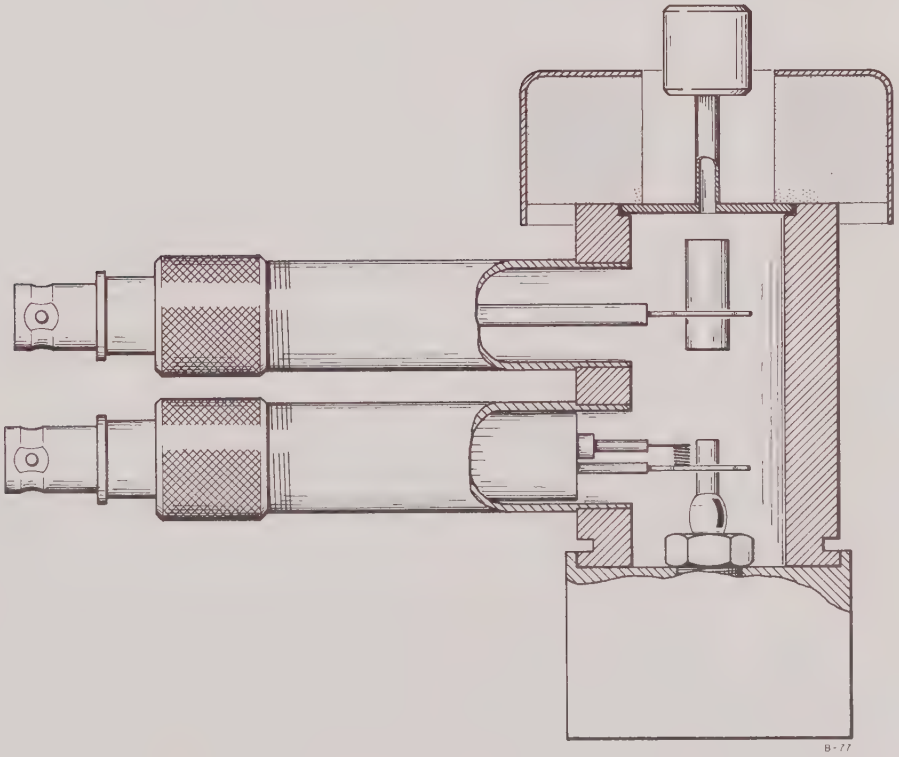



FIGURE 2-2 FID DETECTOR CROSS-SECTIONAL VIEW

# SECTION 3
# OPERATION

## 3.1 GENERAL

This section will cover operation of the FID. However, before going into operational details, a brief explanation of the FID operating principles will be presented.

## 3.2 OPERATING PRINCIPLES

Highly sensitive to organic compounds, the flame ionization detector permits routine analysis of components in the nanogram ($10^{-9}$) range. This detector is insensitive to inorganic compounds and uneffected by small temperature changes. As shown in Figure 2-2, the flame ionization detector consists of a flame tip assembly attached to the detector base. Located above the flame tip is the collector (anode) electrode. Attached to the flame tip assembly is the cathode probe and ignitor coil. The flame is ignited by applying a voltage to the ignitor coil. Ionization voltage is applied via the cathode probe.

Three gas streams are required for operation of the flame ionization detector: Carrier gas, hydrogen, and air. The column effluent mixes with hydrogen and is burned in an atmosphere of air. When organic matter is burned in the hydrogen flame, negative ions and electrons are generated and collected on the anode, thus producing an electrical current in proportion to the amount of material burned. This current is amplified by an electrometer and applied to the strip-chart recorder for representation as a peak on the chart.

- **Detector Selectivity**

The FID responds to most compounds with the exception of those listed in Table 3-1. Note from Table 3-1 the lack of detector response to $H_2O$, $CS_2$, and air. The lack of response to $H_2O$ makes the detector especially suitable for analysis of aqueous samples, biological materials while the lack of response to air makes the detector suitable for analysis of air pollutants. Similarly, the lack of response (no solvent peak) to $CS_2$ makes $CS_2$ an excellent solvent for use in the detector.

TABLE 3-1
COMPOUNDS GIVING MINIMAL OR NO RESPONSE WITH THE FID

| | | |
|---|---|---|
| He | $CS_2$ | $NH_3$ |
| Ar | COS | CO |
| Kr | $H_2S$ | $CO_2$ |
| Ne | $SO_2$ | $H_2O$ |
| Xe | NO | $SiCl_4$ |
| $O_2$ | $N_2O$ | $SiHCl_3$ |
| $N_2$ | $NO_2$ | $SiF_4$ |
| HCHO | HCOOH | |

- **Detector Response vs Flow Rate**

Detector performance depends on the proper choice of gas flow rates, i.e. carrier gas, hydrogen, and air. In general, good sensitivity and stability with 1/8-inch columns are obtained with a mixture ratio, for a first approximation only, of 30/30/300 ml/min respectively, of carrier gas/hydrogen air.

Figure 3-1 demonstrates the relation between detector sensitivity and hydrogen flow rate. Using a 30 ml/min flow of carrier gas and 300 ml/min flow of air, the detector's response for n-heptane reaches a maximum of 30 ml/min of hydrogen. It is obvious that the detector's sensitivity is influenced by the hydrogen flow rate. Individual detectors should be calibrated for maximum response.

As shown in Figure 3-2, air flow rates also affect detector sensitivity. Generally, any flow rate between 250 and 400 ml/min is satisfactory when sample concentrations are in the pg range. With larger sample concentrations, i.e, samples in the $\mu$g range, air flow rates above 300 ml/min may be necessary. Also, the formation of $SiO_2$ from, for example, the use of SE-30 columns can be reduced with increased air flow rates. If necssary, air flow rates can be increased to 1000 ml/min but with an increase in background noise.

## 3.3 FID OPERATION

Before operating the FID make sure the detector is installed on the appropriate detector base(s) in the

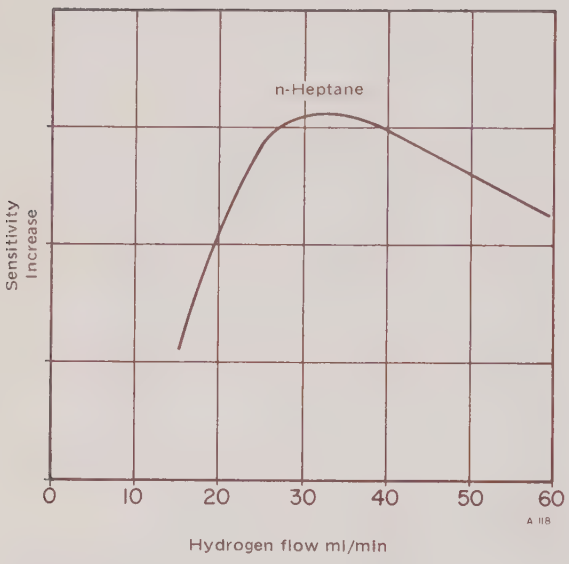



FIGURE 3-1 F.I.D. SENSITIVITY Vs $H_2$ FLOW

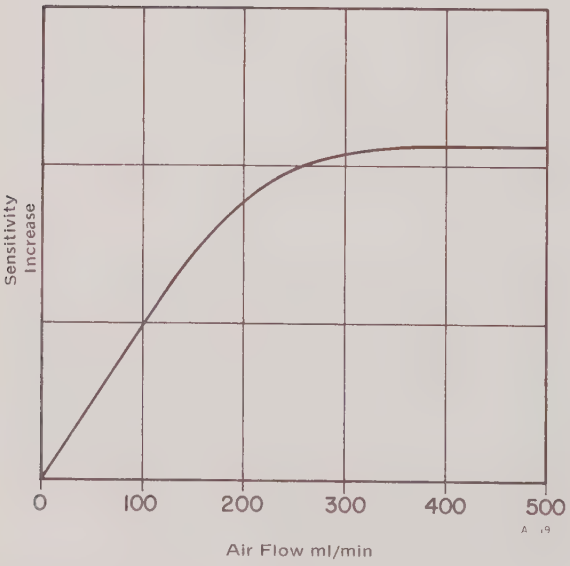



FIGURE 3-2 F.I.D. SENSITIVITY Vs AIR FLOW

basic instrument and that the signal and cell/ignition voltage cables (refer to basic instrument manual for location of cables) are connected to the detector probes. Then, operate detector as follows:

1) Be sure to read all of paragraph 3.2. This paragraph discusses the effects of gas flow rate changes on detector performance and suggests a starting point for setting up the gas flow rates in the detector.

2) Adjust carrier gas, hydrogen, and air flow rates in detector to desired value.

   On *2740, 2840, and 2860* Series instruments, refer to Pressure Gauge or Rotameter Flow Control Module manuals for appropriate settings to achieve desired carrier gas flow rates. Adjust hydrogen and air fine metering valves on flow control panel for appropriate settings to achieve desired hydrogen and air flow rates.

   On 1400 Series instruments, refer to accessory panel for location of carrier gas, hydrogen, and air flow adjusting controls. Set controls for desired detector gas flow rates.

   The calibration and set up of gas flow controls are covered in separate manuals dealing with the appropriate module or instrument basic manuals. Refer to these manuals for this information.

   It is necessary to monitor the flow rate of the gases at the detector when calibrating gas flow controls (except instruments equipped with flow diverters). Refer to paragraph 3.4 for discussion on monitoring gas flow rates at detector.

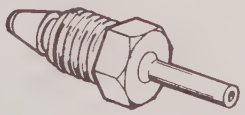
3) Ignite flame in detector via the Electrometer. Select mode of operation and adjust operating parameters with Electrometer. Refer to Electrometer Module manual on operating the flame detector electrometer combination.

### 3.4 MEASURING GAS FLOW RATES AT DETECTOR

Carrier gas and hydrogen exit from the flame tip and air via two holes in the detector mounting nut. Measure gas flow rates as follows:

• Air

Air flow measurements are made with a standard detector base plug supplied with the basic instrument (1400, 2740, 2840 and 2860 Series instruments). The plug blocks off hydrogen and carrier gas but permits the passage of air out the end of the plug. The detector tower and flame tip assembly must be removed and the plug installed on the detector base before the air flow measurement can be made. The detector base plug required is shown in Figure 3-3.



**Cap off ionization detector bases with detector base plug. Plugs are installed when basic instrument is shipped from factory.**

FIGURE 3-3 DETECTOR BASE PLUG

The air flow can be measured by slipping a piece of tubing over the tube extending from the detector base plug. A soapbubble flowmeter (supplied with basic instrument) can be used to measure the air flow rate but with reduced accuracy. The water displacement method is more accurate for the high (250 to 400 ml/min) air flow rates encountered. A good starting point for air flow is 300 ml/min. The air flow can be further adjusted to optimize detector performance after the detector is placed into operation. Refer to paragraph 3.2 for discussion on effects of air flow rate on detector performance.

NOTE

**Be sure to remove the detector base plug and reinstall the flame tip assembly and detector tower after completing air flow measurements.**

• Carrier Gas

Carrier gas flow measurements are made at the end of the flame tip assembly. As shown in Figure 3-4, a .160" O.D. tube must be used to slip over end of flame tip. The other end of the tube connects to a soapbubble flowmeter (soapbubble flowmeter supplied with basic instrument). Use soapbubble flowmeter to measure carrier gas flow through detector. Operation of flowmeter is described in the basic instrument manual. Note that the tower cap must be removed before the .160" O.D. tube can be connected to the flame tip.

**WARNING**

**To avoid a shock hazard, make sure detector cell voltage is turned off at Electrometer and that the cell voltage cable is disconnected from the tower probe.**

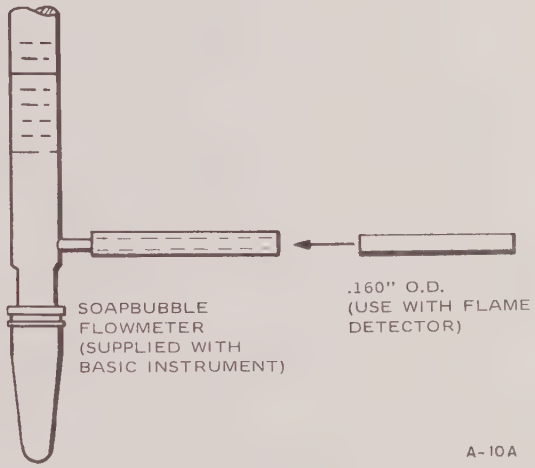



FIGURE 3-4 SOAPBUBBLE FLOWMETER SETUP

A good starting point for carrier gas flow is 30 ml/min; however, the carrier gas flow should be further adjusted to optimize column efficiency after placing the detector into operation. Since the proper flow of carrier gas through the column/detector system is important, we recommend you obtain a copy of the Varian Aerograph Technical Bulletins No. 124-66 on Column Technology and No. 126-66 on Flame Detectors for additional information on this subject.

• Hydrogen

Hydrogen flow measurements are made in the same manner as carrier gas measurements, i.e., connecting the .160" O.D. tube to the flame tip using the

soapbubble flowmeter to measure the hydrogen flow rate. A flow rate of 30 ml/min is recommended as a starting point. The hydrogen flow rate can be changed to optimize detector performance after the detector is placed into operation. Refer to paragraph 3.2 for further discussion on effects of hydrogen flow rate on detector performance.

If the carrier gas flow was previously set up, you will be measuring the combined flow of carrier gas and hydrogen. Therefore, hydrogen flow is equal to the difference between carrier gas flow rate and total flow rate.

# SECTION 4
# SERVICING

## 4.1 GENERAL

This section of the manual discusses routine maintenance and cleaning procedures required to keep the flame detector working properly.

## 4.2 MAINTENANCE AND CLEANING

Column materials sometime leak into detector components causing contamination. To remove contaminants that build-up on the detector components, proceed as follows:

1) Turn off cell voltage at Electrometer.

2) Remove flame detector to be cleaned as outlined in paragraph 2.7.

3) Remove probes from tower. Be sure to remove collector cylinder before removing collector probe.

4) Place probes in ultrasonic cleaner containing water. Clean probes for approximately 30 minutes, then air dry. If an ultrasonic cleaner is not available, wipe off probe elements with ether.

5) Place detector components, i.e., flame tip assembly, collector cylinder, etc., into ultrasonic cleaner containing water. Clean components for approximately 30 minutes, then air dry. If ultrasonic cleaner is not available, clean components with acetone.

6) Wipe off detector tower and cap with acetone.

7) Reinstall detector as outlined in paragraph 2.1.

NOTE

**If FID requires cleaning the detector base must also be cleaned. Refer to detector base cleaning procedures in the basic instrument manual.**

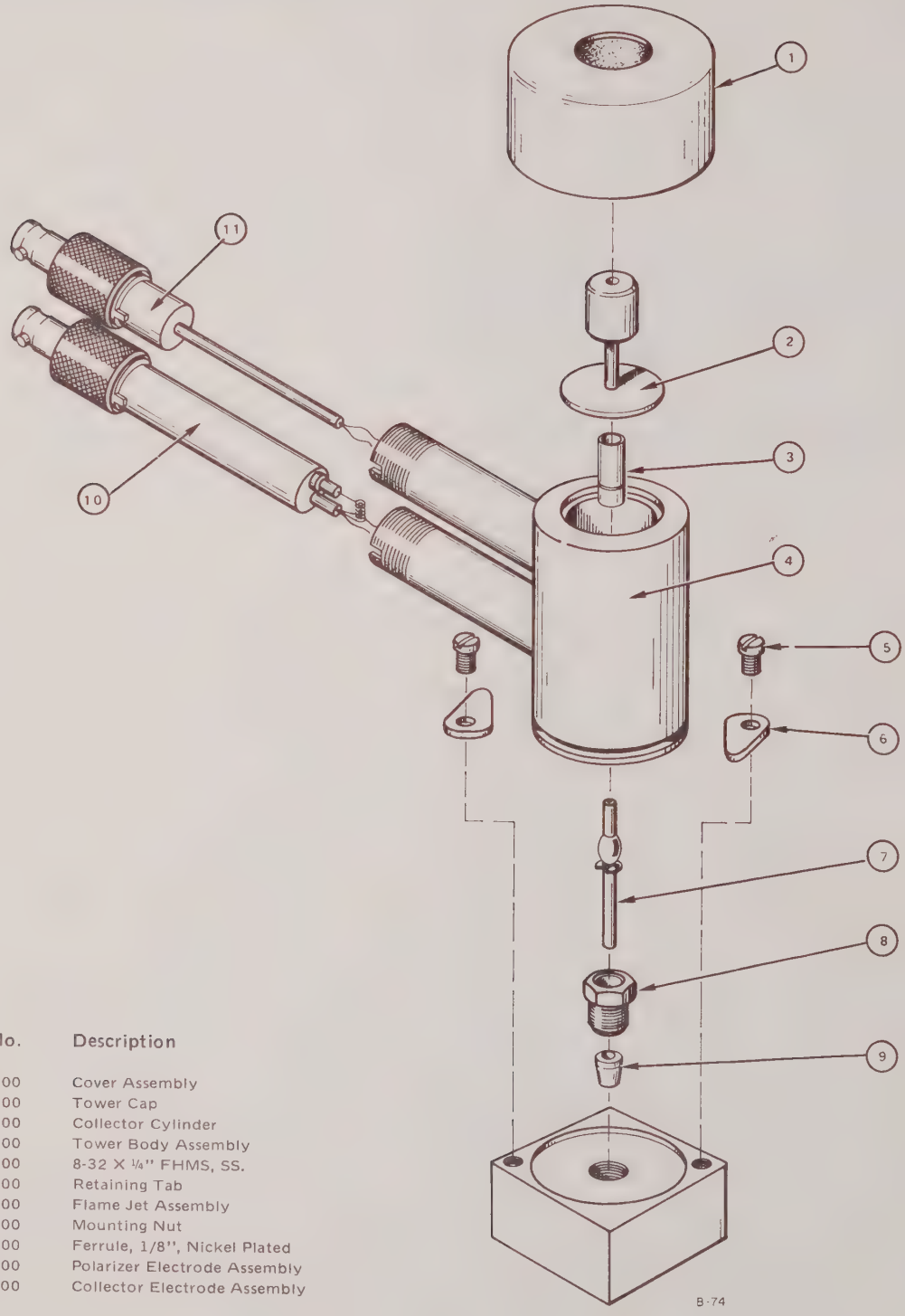
# SECTION 5
# ILLUSTRATED PARTS LIST

## 5.1 GENERAL

This section of the manual contains the illustrated parts list for the FID detector. See title and page number below for location of drawing.


DRAWING TITLE — PAGE NO.

FID Hardware Location Drawing ..... 5-2




| Item | VA Part No. | Description |
|---|---|---|
| 1 | 01-000693-00 | Cover Assembly |
| 2 | 02-000594-00 | Tower Cap |
| 3 | 32-000021-00 | Collector Cylinder |
| 4 | 01-000658-00 | Tower Body Assembly |
| 5 | 15-004722-00 | 8-32 X ¼" FHMS, SS. |
| 6 | 08-000072-00 | Retaining Tab |
| 7 | 02-000607-00 | Flame Jet Assembly |
| 8 | 15-001207-00 | Mounting Nut |
| 9 | 16-000389-00 | Ferrule, 1/8", Nickel Plated |
| 10 | 02-000957-00 | Polarizer Electrode Assembly |
| 11 | 02-000955-00 | Collector Electrode Assembly |

FID HARDWARE LOCATION DRAWING

varian
instrument division
611 hansen way / palo alto / ca 94303 / u.s.a. / phone 415-493-8100

# AEROGRAPH®

# $Sc^3H$

# ELECTRON CAPTURE DETECTOR

# INSTRUCTION MANUAL

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Manufactured in the United States of America

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# SECTION 1
# INTRODUCTION

## 1.1 GENERAL

This instruction manual describes the Aerograph® Scandium Tritide Electron Capture Detector ($Sc^3H$ ECD). The primary feature of this electron capture detector is that it permits high temperature operation from a tritiated radioactive source. A foil temperature up to 325°C may be employed if chromatographic conditions require it. Additional advantages of the detector are as follows:

- High Sensitivity—Much greater sensitivity than $Ti^3H$ and $^{63}Ni$ ECD at conditions described in Section 3, Detector Principles and Characteristics.
- Low Dead Volume—The 0.3 ml internal cell volume is designed for compatibility with capillary column applications.
- Rugged Construction—The ceramic insulators have been designed for high strength.
- Detector Interchangeability—With the Aerograph universal detector base, the scandium ECD can be rapidly interchanged with the FID or AFID.
- Readily Accessible Foil—In less than a minute, the foil can be removed for cleaning or replacing.

## 1.2 SUMMARY OF MANUAL SECTIONS

Included below is a description of the seven additional sections of this instruction manual:

**Section 2 Simplified Instruction Sheet**
Abridged setup and operation instructions for experienced chromatographers familiar with electron capture detector operation, particularly Aerograph detectors. This one-page instruction sheet will also be useful as a periodic checklist for less experienced chromatographers. For those less familiar with ECD operation, a good understanding of Section 3 is highly recommended.

**Section 3 Detector Principles and Characteristics**
Although the physical apparatus of an electron capture detector is basically very simple, the operating characteristics are quite complex as shown by the list below.

**3.1 General**
**3.2 Definition of Terms**
**3.3 Principles of Operation**
**3.4 Standing Current vs. Voltage**
**3.5 Standing Current vs. Temperature and Pressure**
**3.6 Standing Current vs. Contamination**
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**3.11 Radioactive Emanation**
**3.12 Summary of Optimum Conditions**

**Section 4 General Installation**
General instructions for detector installation.

**Section 5 Individual Instrument Installation and Parts List**
Special installation information for specific Aerograph instruments.

**Section 6 Operation**
Describes GC instrument controls and how they are set for ECD operation.

**Section 7 Troubleshooting**
Lists commonly observed operational problems and their corresponding remedies.

**Section 8 Routine Maintenance**
Discusses periodic tasks which should be performed to obtain best possible performance.

## 1.3 SPECIFICATIONS

**DETECTIVITY**
1.0 pg Lindane (standard conditions)
0.1 pg Lindane (optimized condition, see Section 3)

**LINEAR RANGE**
1000

**NOISE LEVEL**
$\sim 10^{-11}$ amp (3% @ 4 x $10^{-10}$ amps full scale)

**UPPER LIMIT TEMPERATURE**
325°C

**SOURCE**
1000 mCi $^{3}H$

**CELL VOLUME**
0.3 ml

# SECTION 2
# SIMPLIFIED INSTRUCTION SHEET

## 2.1 GENERAL

This section summarizes, in short form, general instructions for installation of the Electron Capture Detector in Aerograph® Instruments.

## 2.2 INSTALLATION (in preconditioned GC)

1) Shut off column flow rate to avoid contaminating the foil with column bleed when the cool cell is installed.

2) Mount cell in detector base without foil holder installed (special hollow shaft nut driver included).

3) Mount tower over cell; center the cell to avoid shorting to tower; clamp securely to base (base to tower mechanical contact must be clean for best heat transfer).

4) Insert electrodes in tower arms (make sure that probe clips are not shorted to ground).

5) Position thermocouple in thermocouple (T.C) well on top of tower. Install foil holder and foil. Tighten cell cap.

6) Align exit tube and/or cell to prevent grounding of voltage or signal probes, and tower cap.

7) Place tower cap over exit tube. Align slot with T.C. well.

8) Mount plastic vent tube on cell exit tube to fume hood (optional).

9) Pack insulation around tower to hold in heat.

10) Attach electrometer cables (voltage, input).

11) Heat detector to about 50°C higher than column temperature. Retighten tower mounting screws.

12) Turn on column flow.

13) Precheck of system with a cold column, or a blank tube in place of a column, is a recommended pre-operational standing current check.

## 2.3 OPERATION

1) Carrier flow rates between 30-40 ml/min are recommended; however, as low as 10 ml/min is usable.

2) A 30 ml/min, $N_2$ purge is often helpful in minimizing foil contamination problems (can be added at $H_2$ port).

3) Heat foil only hot enough to minimize foil contamination; 300°C and hotter should be avoided, when possible, to extend foil longevity.

4) Remove and store foil during non-operational times (overnight, weekends, holidays) in labeled storage vial provided.

5) Check standing current regularly to give indication of expected detector performance.

6) Bake out columns longer than usual. Remove column packing from the column segment that extends into the detector base. Higher temperature in this region (typically ~50°C) can cause excessive bleed and possible cell contamination.

7) Operate septum as cool as possible. Use on-column injection with unheated injectors wherever possible.

8) Clean foil with KOH and MeOH using ultrasonic cleaner (foil should be removed from holder to prevent contamination by caustic residues.

**WARNING**

**See paragraph 6.2.6 for full procedure. Foil must not be touched by hand.**

9) Clean cell with acetone or non-conductive solvent when a measurable ceramic leakage is observed with no foil installed (false standing current).

10) A -90 Vdc operation is recommended for minimizing contamination effects.

## 2.4 TROUBLESHOOTING (symptoms, causes, remedies)

1) Low or No Standing Current

   a) Low supply of betas (foil has been overheated or contaminated). Clean foil.

   b) Carrier gas has impurities (install $O_2$ trap—regenerate or replace existing $O_2$ trap).

   c) Contamination from column (recondition at elevated temperature).

   d) No voltage on cell (check continuity). Possible electrode short to ground.

   e) No connection to electrometer input (check continuity).

2) Low Sensitivity

   a) Sample not injected properly (leaky syringe or leaky septum).

   b) Sample lost to column (active sites or absorption).

   c) Low standing current (see paragraph 1).

3) Baseline Instability

   a) Excessive column bleed (recondition column at elevated temperatures overnight).

   b) Insulator leakage (ceramic insulators contaminated). Clean insulators with acetone in ultrasonic bath.

   c) Contaminated foil (remove and clean or replace).

# SECTION 3
# DETECTOR PRINCIPLES & CHARACTERISTICS

## 3.1 GENERAL

Although the physical structure of an electron capture detector is basically simple, the operating characteristics are complex. The operating characteristics are discussed in the following text.

A thorough understanding of this section is not required for those with routine applications, but it is particularly important for those interested in obtaining the optimum performance from the scandium ECD.

A more comprehensive discussion is found in *Gas Chromatography Detectors*, C. H. Hartmann, Anal. Chem., 43, 113A-125A (1971).

## 3.2 DEFINITION OF TERMS

Refer to the following text for the definition of terms used to describe the ECD.

### 3.2.1 Sensitivity

Sensitivity is a measure of detector effectiveness as a transducer in converting the sample into a measurable electrical signal. However, the sensitivity factor (amp-ml/g) of a detector is not a very useful term to the chemist. Sensitivity coupled with the detector noise level however, can be used to determine detectivity. The sensitivity factor should therefore be a constant of the detector. For the ECD there are five conditions which must be satisfied before the sensitivity can be considered a constant. These conditions are as follows:

1) The standing current (for definition, see paragraph 3.3.1, Principles of Operation) should be constant; a condition rarely achieved because of the gradual contamination of the radioactive source.

2) The flow rate should be constant. Since the ECD has a concentration dependency, the higher the flow rate, the lower the sensitivity.

3) The coefficient of capture must be constant. For example, the more halogens bound to the molecules, the greater the sensitivity. This range of coefficients can result in 0.1 ppb detection of heavily halogenated hydrocarbons such as carbon tetrachloride to only 0.1% detection of chlorobenzene, a range of $10^7$.

4) The density of the carrier gas inside the ionization chamber should be constant. Since pressure and temperature affect density, holding these parameters constant, will result in the generation of a constant standing current, resulting in constant capture reactions.

5) The resulting response must be within the linear range of the detector.

For those interested in the sensitivity calculation, the following example is given:

$$\text{Sensitivity} = \frac{\text{Peak Area} \times \text{Flow Rate}}{\text{Sample Weight}}$$

Peak Area from a triangular approximation:

½ Peak Height (amp) x Peak Width (min)

Flow Rate = Volume flow rate at atmospheric pressure (ml/min)

Sample Weight = Amount of constituent injected (grams)

For Lindane Sample under typical conditions:

Peak Height = $10^{-9}$ amp
Peak Width = 1/6 min
Flow Rate = 30 ml/min
Sample Weight = 100 pg
Voltage on Cell = -90 Vdc

Sensitivity:

$$\frac{½(10^{-9}\ \text{amp})(1/6\ \text{min}) \times 30}{(100 \times 10^{-12}\ \text{g})} = 25\ \text{amp ml/g}$$

typical of an optimized system.

### 3.2.2 Noise Level

By convention, the noise level is the peak-to-peak amplitude of fast term recorder oscillations. However, any random baseline disturbance that could be misinterpreted as a peak must be considered in a noise determination. For example, if p,p'DDT is the response of interest, then fast term noise is not likely to be a limiting factor on this 60 second wide peak; however, other disturbances such as temperature fluctuation could cause baseline instabilities that could yield ambiguities in the interpretation of the real GC peak.

A typical example of noise magnitude for an ECD is $10^{-11}$ amps which would be approximately a 3% peak-to-peak recorder response on a 1 mV recorder at an attenuation of $4 \times 10^{-10}$ amps on the electrometer, e.g.,

$$0.3 \times 4 \times 10^{-10}\text{amps} = .12 \times 10^{-10}\text{amps} \sim 10^{-11}\text{amps}$$

**NOTE**

**For those few analysts who require the use of extremely high electrometer sensitivities such as $4 \times 10^{-10}$ amps full scale or higher, a special modification can be made to the electrometer to reduce the noise level by a factor of 3, resulting in a 1 second time constant. This modification can only be performed by a Varian field service representative.**

### 3.2.3 Detectivity

Detectivity is related to the smallest detectable response that can be read above the noise level. Common practice is to declare this smallest readable peak above the noise level as two times the amplitude of the peak to peak noise, i.e., a signal to noise ratio (S/N) = 2.

Although for good quantitative determinations, such a small response, could not garner much confidence, this definition is valuable for comparison purposes in specifications. One method of determining a numerical detectivity is to divide the sensitivity (from above) into twice the noise level:

$$\text{Detectivity} = \frac{2N(\text{amp})}{\frac{S(\text{amp-ml})}{g}}$$

Typical noise level for a $^3H$ ECD is $10^{-11}$ amps. Therefore, Lindane detectivity from the sensitivity expression is as follows:

$$\text{Detectivity} = \frac{2 \times 10^{-11}\text{ amps}}{25\text{ amp ml/g}} = 8 \times 10^{-13}\text{ g/ml}$$

The previous two definitions have greatest utility when specifying the detector performance. The chemist, however, simply wants to know how small a sample he can detect. A practical definition of detectivity is the minimum detectable quantity, or MDQ, and is that amount of sample that will yield a response two times greater than the noise level. The units of MDQ are usually expressed as sample weight.

### 3.2.4 Linearity

Linearity is the range of sample concentrations where proportionality exists between sample concentration and detector response. There are two limits to the linearity curve. The lower concentration limit is set by the limit of detection and the upper limit is sent by an arbitrary percentage deviation from the linearity curve, usually about 5%. The importance of detector linearity is that it alerts the analyst to concentrations for which he can be confident of a constant detector calibration factor. A detector with a small range of linearity such as the ECD can have a useful calibration range with a much greater range than the linear range. Precision of determination is diminished in the non-linear regions, so the analyst should proceed with caution. Linearity of the detector will be defined as the ratio of the highest sample concentration (just within the proportional response region) to the smallest detectable sample. An example for the Sc$^3$H ECD for the sample Lindane is as follows:

$$\text{L.R.} = \frac{\text{Weight of sample to yield response within 5\% of calibration curve}}{\text{Weight of sample detectable at two times noise level}}$$

$$= \frac{0.1\text{ ng}}{0.1\text{ pg}} = 1000\text{ fold}$$

## 3.3 PRINCIPLES OF OPERATION

The scandium ECD operation is based on the principle that certain molecules have the characteristic of being able to absorb or capture free or low energy electrons. A steady supply of free electrons is generated and collected within the detector. This supply of free electrons will subsequently be depleted when a capturing species enters the detector. The amount of decrease in the free electron population is not only a measure of the amount of capturing species that pass through the detector, but also a measure of the degree of capturing ability (capture coefficient) of that particular species. Capture coefficients have a very wide range of variability allowing carbon tetrachloride to be detected at a tenth of a part per billion on the low end to only one thousand parts per million detectability of chlorobenzene at the high end.

### 3.3.1 Details of Operation

Figure 3-1 shows a schematic cross section of the concentric electrode electron capture detector, US Patent No. 3,277,296. The upper region or cathode of the detector consists of a 1/4" OD, 3/16" ID Kovar tube which serves as an ionization chamber. A 1/2" x 1/2" x .005" radioactive source is coiled in the form of a cylinder and placed inside the Kovar tube. As a carrier gas ($N_2$) passes through the ionization chamber, the beta particles emitted from the radioactive source ionize the nitrogen, releasing free electrons as described in the following equation:

$$\beta + N_2 \rightarrow N_2^+ + e^- + \beta$$

Each beta particle ionizes approximately 10 nitrogen molecules before its energy is expired. Although the foil has been charged to approximately 1 Ci only a portion of this total is available at the surface for nitrogen carrier ionization. Since the ionization chamber is charged with a negative potential, the free electrons formed are quickly collected counter current to the $N_2$ flow at the anode which is upstream from the cathode. This presents ideal separation of ionization products—the $N_2^+$ being swept out of the ionization chamber by the carrier gas and the free electrons being repelled upstream by the polarization voltage. The total current generated by the free electrons is a steady current background or standing current of a reasonably high magnitude compared to other GC detectors, e.g., it can be as high as $4 \times 10^{-8}$ amps in an ideal GC with no column bleed. This is referred to as a standing current of the ECD.

$\Sigma e^- = (1\text{-}4) \times 10^{-8}$ amp standing current

An operational GC can yield up to $3 \times 10^{-8}$ amp, yet can still be operational as low as $1 \times 10^{-9}$ amp.

As an electron capturing species enters the detector ionization chamber, it encounters this high density electron flow and a decrease in standing current is observed as the electrons are gathered up by the sample molecules (S).

> $S + e^- \rightarrow$ ion products with various charges which depend on whether the process is associated or disassociative.

This decrease in standing current is measured with the electrometer as a quantitative measure of the substance.

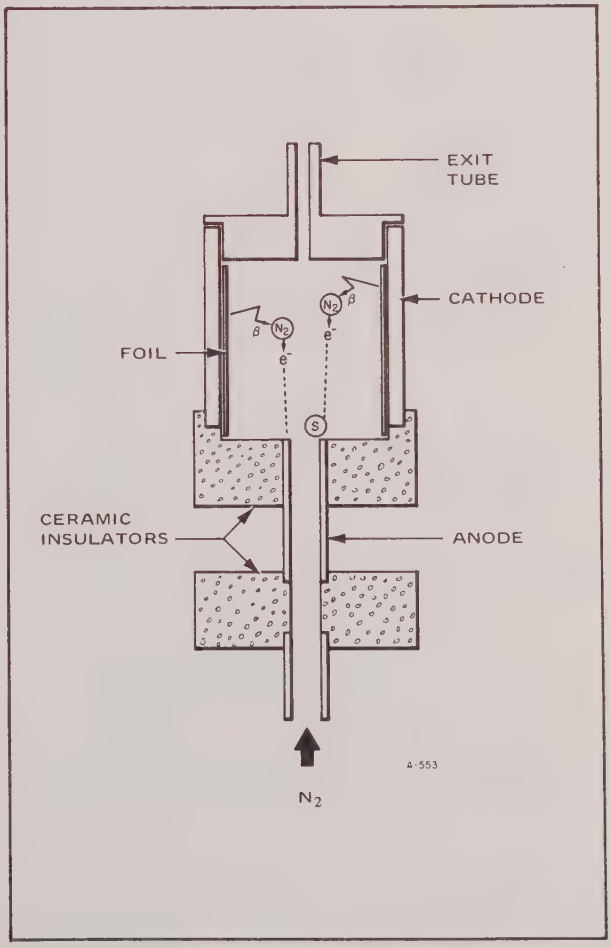



FIGURE 3-1 $Sc^3H$ SCHEMATIC OF CONCENTRIC TUBE ECD

### 3.4 STANDING CURRENT/CELL VOLTAGE

The voltage difference at which the anode most readily collects the bulk of the free electron current is the potential for optimum ECD performance. This voltage for a clean detector can be determined from a plot of standing current vs. detector voltage as shown in Figure 3-2. Since the Aerograph electrometers do not provide a variable voltage source, it is not expected that the chemist could reconstruct the full curve shown in Figure 3.2. Based mostly on contamination considerations, -90 Vdc is the recommended operating potential. The solid curve of Figure 3-2 is what would be expected from a new foil and clean cell and an "ideal" GC system.

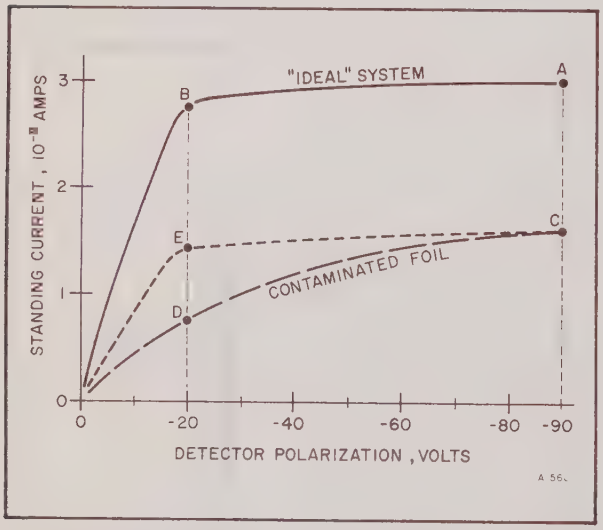



FIGURE 3-2 STANDING CURRENT RELATIONSHIPS

The "ideal" GC system is ideal only from detector considerations and is described as follows:

1) Clean piece of metal tubing connecting injector outlet to detector inlet, i.e., no bleed from column liquid phase. This ideal can be achieved with some chromatographic separation where gas solid partitioning is employed.

2) Blank off plug on injector inlet, i.e., no bleed from septum.

3) Oxygen trap (VA Part No. 86-000056-00) employed in the $N_2$ line, i.e., $O_2$ captures electrons and hence will decrease detector performance.

4) All pneumatic joints are sealed to prevent any air from leaking into the carrier stream, i.e., a sealed system should hold 60 psig for at least one hour.

With an "ideal" system and about an hour of system purging, the solid curve in Figure 3-2 will be obtained with the "knee" of the curve at about -20 Vdc. The ratio of standing current of B to A should be 0.85 or greater (usually between 0.85 and .90). If the system cannot pass this test, poor performance can be expected to closely approach a 0.80-0.85 value. However, most analyses are performed under less than "ideal" conditions. This results not only in a deterioration of the standing current (A to C) but in a noticeable change in the shape of the voltage curve as shown by the dashed line in Figure 3-2 where the ratio of D to C can be from 0.4 to 0.6 or so. Depending on other chromatographic indicators such as solvent and peak responses, the foil might have to be cleaned or replaced as will be described more fully in later sections. A clean but depleated foil would yield a Voltage/Standing Current curve depicted by the short dashed line of Figure 3-2 where again the ratio of the "knee" current to plateau current is about 0.85 (E/C).

### 3.5 STANDING CURRENT VS. TEMPERATURE AND PRESSURE

The standing current increases somewhat with the increased density of the carrier gas, i.e., the more compressed the $N_2$ molecules are within the ionization chamber, the greater the possibility of bombardment with the beta particles and, therefore, the greater the net ionization. As the temperature is increased inside the detector, the density will decrease tending to decrease the standing current. Also, higher temperatures increase the capture coefficient of any $O_2$ impurity concentration in the $N_2$ carrier which will decrease the standing current even further. The resulting change in standing current in an absolutely clean system would not be greater than 10% with the detector temperature change from 100° to 300°C. If the pressure within the chamber could be increased, the density would increase and, hence, the standing current would increase. However, the increased pressure for the same mass flow rate would slow down the purge rate through the cell, and therefore, the contamination effect would tend to increase. This discussion is presented mostly for the sake of interest since

the detector temperature is set mostly by chromatographic conditions (somewhat hotter than the column temperature to minimize contamination from column effluent) and foil life expectancy (the cooler it operates, the longer the tritium lasts).

The effects of temperature on standing current assume that as the temperature is increased, the activity level of tritium on the foil remains constant. With a sustained temperature above 200°C, this assumption is not valid. The tritium will be released from the $Sc^3H$ foil at ever increasing rates as the temperature is increased. The maximum rate (by license requirements) that will ever be released is 10 μCi/min @ 325°C, and then only when the foil is brand new; 10 μCi/min is less than the rate of the $Ti^3H$ foil at 225°C. Paragraph 3.12 has a complete discussion of $^3H$ emanation. As the tritium is released, the standing current decreases, but fortunately not proportionally. The standing current and $^3H$ release have been monitored at several temperatures for 60 day periods in an "ideal" system (see paragraph 3.3). The results of standing current decay are shown in Figure 3-3. The standing current during the first 30 days at 325°C dropped 21% for the new foil; only 9% additional decrease for the second 30 day period.

One might conclude that with a standing current drop of 21% in the first 30 days, the amount of $^3H$ released would be about 21%. Actually, the total $^3H$ removed from the foil was less than 2% of the total foil loading. One explanation for this apparent anomaly is that most of the tritium that is loaded onto the foil is "buried" in the substructure of the scandium substrate and never becomes available for ionizing the $N_2$ carrier gas. Another conclusion one might reach is that a much lower total activity foil might be just as effective as 1000 mCi foil. This is, in fact, the case if standing current is the only concern; e.g., a 100 mCi foil will generate a satisfactory standing current. High temperature stability of $^3H$ is of primary interest; and the thicker the Scandium layer, the greater the temperature stability. The foil is overloaded with $^3H$ so good high temperature stability is achieved.

In summary, even with an ideal system (no column bleed), the standing current will be expected to decay somewhat with time and high temperatures.

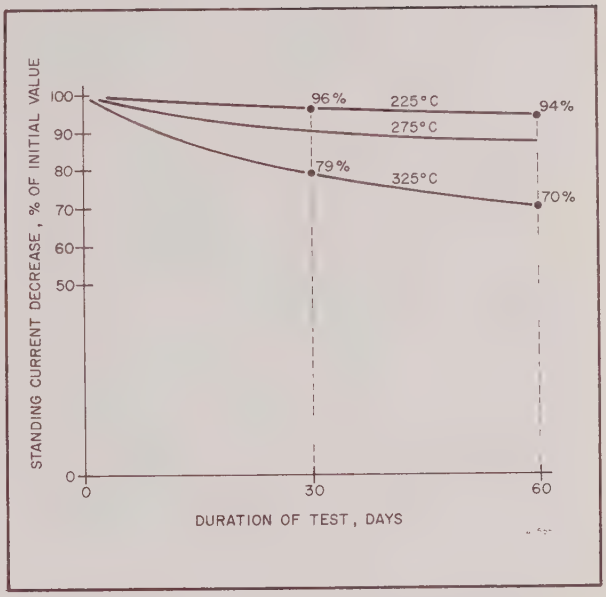



**FIGURE 3-3 STANDING CURRENT DECAY WITH TIME & TEMPERATURE (IDEAL SYSTEM)**

## 3.6 STANDING CURRENT VS. CONTAMINATION

As the radioactive foil ages from chromatographic usage, the standing current will tend to decrease even further. A decrease in standing current is caused by any one or a combination of the following:

1) Fewer beta particles, i.e., loss of $^3H$ radioactivity because of sustained 325°C operation.

2) Poor quality nitrogen, i.e., carrier gas contaminated with $O_2$ or column effluent products, or not enough $N_2$ flow.

3) Faulty potential applied to collect current, i.e., possibly grounded out.

4) Faulty electrometer connection to measure current, i.e., possibly shorted cable or open connection to cell.

Of the above-mentioned factors, the most prevalent and least controllable is the column effluent contamination. In the early years of the electron capture detector, it was believed that the $Ti^3H$ foil contaminated (lost standing current) because it could not be safely heated above 225°C. With most

pesticide column operating temperatures about 200°C, this 25°C temperature differential was believed to be insufficient to prevent condensation on the foil surface. This condensation theory became questionable with the advent of the $^{63}Ni$ foil with a 400°C upper temperature limit. Even at 400°C, the $^{63}Ni$ ECD suffers a gradual loss of standing current. Since $^{63}Ni$ is not likely to vaporize at 400°C, the loss of standing current must be due to some form of foil contamination (not likely condensation, but possibly absorption or an active surface for sample nucleation).

Even today, a completely comprehensive explanation is not available for what causes these weak beta emitters to contaminate. The optimum temperature under a wide variety of operating conditions has not been evaluated. Nonetheless, the guidelines of 50°C hotter than the column temperature seems to be about optimum.

In an attempt to test the minimum contamination condition of the $Sc^3H$ ECD, a sustained and continuous 325°C operation was undertaken with over 250 pesticide extracts being analyzed during a 30 day period. The results of the standing current decay is shown in Figure 3-4. A 21% decrease is noted which is the same amount of standing current loss with the "ideal" system of paragraph 3.3. Therefore, it is concluded that the standing current loss from these actual analytical conditions is not due to foil contamination, but rather due to $^3H$ release. Of course, another conclusion is that a lower foil temperature could be used to keep the foil from being contaminated and thereby considerably extending the life expectancy of the foil. A foil temperature between 250°C and 275°C is estimated to be a good compromise between foil contamination and foil $^3H$ depletion for typical pesticide columns operated at 200°C.

### 3.7 SENSITIVITY VS. CONTAMINATION

Since ECD sensitivity is heavily dependent on the standing current, a similar effect of usage on sensitivity (as with standing current in paragraph 3.6) could be expected. Surprisingly, the sensitivity is not as adversely affected by contamination (at -90 Vdc) as the standing current. Whereas some increase in sensitivity (no known explanation) is noted during the first 10 days, a 10% decay in sensitivity is observed in the last 20 days of the test period (as shown in Figure 3-5). Poor sensitivity, besides being caused by low standing current, will also depend on these factors.

1) Inertness of the GC system (solid support, minimum metal surfaces, etc.)

2) Type of liquid phase used in the column.

3) Types of sample extracts being analyzed.

4) Temperature of the foil.

5) Amount of $N_2$ purge gas being used.

Guidelines are being presented in this manual for best procedures to follow, but in no way should these be taken absolutely.

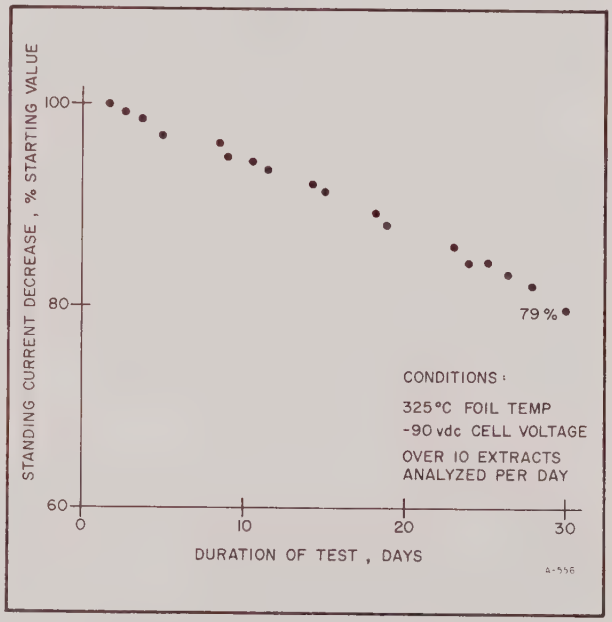



**FIGURE 3-4 STANDING CURRENT DECAY UNDER ANALYTICAL CONDITIONS**

### 3.8 SENSITIVITY/FLOW RATE

A certain minimum flow rate of $N_2$ through the ionization chamber is required to prevent back diffusion of $O_2$ from the air via the exit tube. Slight standing current loss is observed at 10 ml/min and a 50% loss is observed at 5 ml/min. Because of the ECD's concentration type characteristics, the higher the flow rate, the lower the sensitivity. This is shown in Figure 3-6 where make-up $N_2$ at various flow rates, is added to the FID $H_2$ port that "tees" into the detector base. When some decrease in sensitivity can be tolerated, make-up gas has been shown to be advantageous in minimizing contamination of the foil.

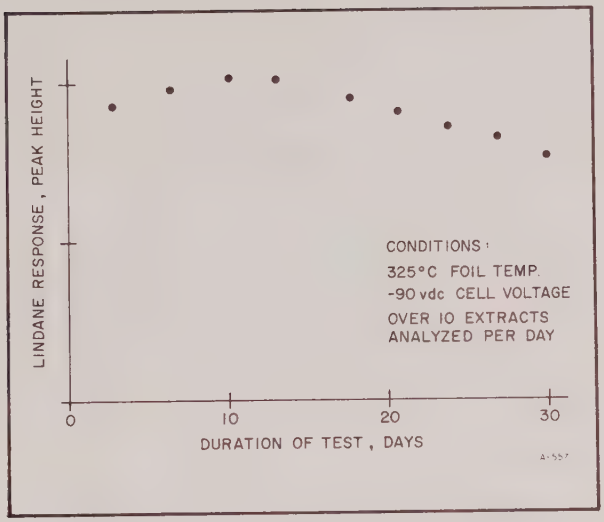



FIGURE 3-5 SENSITIVITY DECAY UNDER ANALYTICAL CONDITIONS

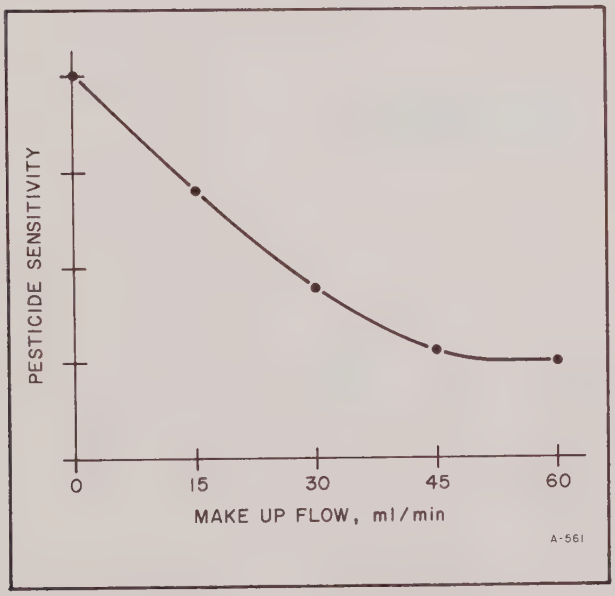



FIGURE 3-6 SENSITIVITY DECAY WITH $N_2$ FLOW MAKE UP

## 3.9 CAPTURE SENSITIVITY/TEMPERATURE

The temperature of the ionization chamber will sometimes have an influence on the capture coefficient of the sample molecule. Some data is shown in the two figures of Figure 3-7 for three chlorinated pesticides and for four organophosphorus pesticides. Where contamination considerations permit it, and where foil life expectancy will allow it, there might be some advantage in selecting a detector temperature for best sensitivity if that is a concern. This section should indicate to the analyst the importance of calibrating the ECD at the same detector temperature that the actual samples are to be analyzed.

## 3.10 CHROMATOGRAPHIC CONDITIONS

Although the analyst usually has little choice but to use gas-liquid partitioning to accomplish his separations, there are a few precautions that can be taken to minimize column bleed.

1) Extra column conditioning: One of the common pesticide column packings is 2.5% DC-200 and 2.5% QF-1 liquid phase on Gas Chrom Q 100/120 mesh. Normally a 16 hour (overnight) bakeout at 245°C is adequate, but a 64 hour (over a weekend) bakeout at 245°C is even better.

2) The column packing protection: The column packing should not protrude into the detector base region where hotter temperatures are used to heat the radioactive foil. These hotter temperatures will tend to strip the liquid phase from the solid support and then this stripped off material would contaminate the foil.

3) On-column injection procedure: This allows a cooler operating injector and, hence, a cooler operating septum which minimizes standing current decrease due to septum bleed. A septum stand-off called an Injector Port Extender (Part No. 16-000665-00) is highly recommended.

4) Column absorption: It is not uncommon for one or more of the pneumatic passages (injector, column, detector) to cause some sample molecules to become irreversably lost, e.g., decomposed, absorbed, or polymerized. The pesticide p,p'DDT is one of the most notorious examples. For the best quantitative confidence, an inertness check should be made on the GC system. This is simply performed by injecting a series of six or more different sample weights at the low end of the calibration or linearity curve. The smallest weight should be at least as small as five times the noise level. The extension of the "best fit"

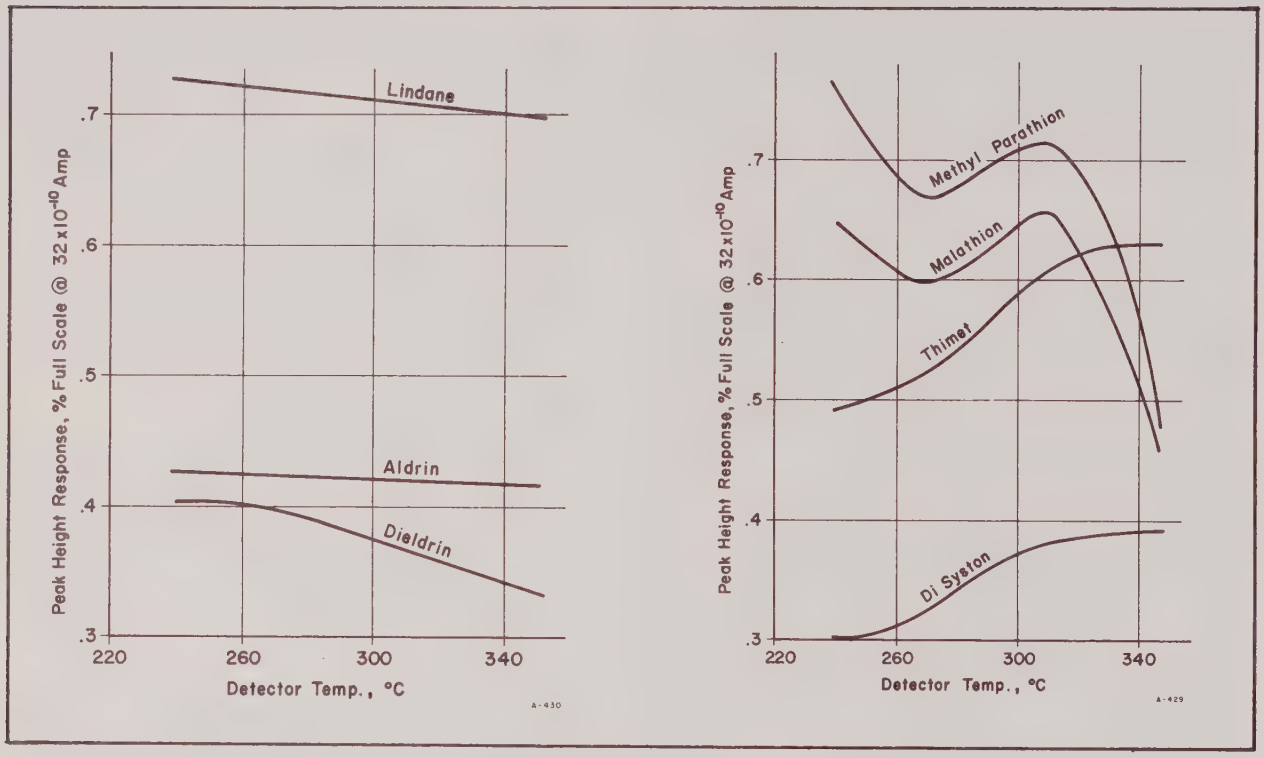



FIGURE 3-7 CAPTURE SENSITIVITY vs. TEMPERATURE

curve should intersect the origin of a cartesian coordinate plot. If the curve bends toward the sample weight axis, sample is being held up by the GC (see Figure 3-8).

### 3.11 RADIOACTIVE EMANATION

In 1963, G.R. Shoemake, J.E. Lovelock, and A. Zlatkis published a piece of work in *J. Chromatography*, Volume 12, Page 316, showing that $^3H$ is released from tritiated sources used in gas chromatographic detectors. The early report was quite limited on statistical information and the time-temperature characteristic of this application, therefore a great deal of new data was needed. As a part of the requirements for obtaining a $Sc^3H$ license, it had to be shown that the new radioactive source was at least as stable at 325°C as the previous $Ti^3H$ source was at 225°C. After extensive testing of $Ti^3H$ foils, it was determined that the average or typical $^3H$ release rate (emanation rate) for 250 mCi $Ti^3H$ foil at 225°C was approximately 10 μCi/min. Experimental methods and test procedures were verified with a collaborative program with the Isotope Division of the Oak Ridge National Laboratory (ORNL). The agree-

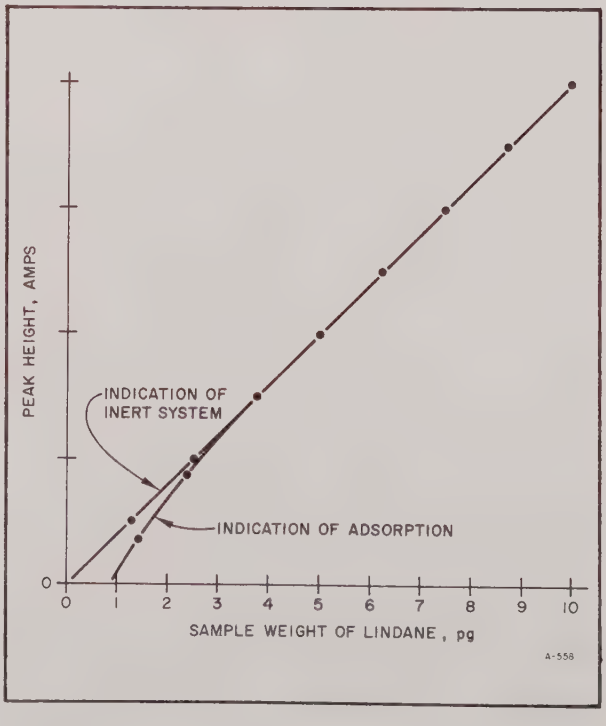



FIGURE 3-8 GC INERTNESS CHECK

ment in results was extremely close for such a sensitive determination. After similarly testing the Aerograph processed 1000 mCi $Sc^3H$ foils at 335°C, an average emanation of 5.5 μCi/min, was found. This is almost a factor of two less emanation than the actual amount from previous $Ti^3H$ results. As an additional safety factor, a 325°C upper temperature limit for the $Sc^3H$ ECD was recommended. This is a conservative operating condition which yields less radioactive release than the previously approved industry standard, i.e., the $Ti^3H$. The lower the operating temperature, the lower the emanation, e.g., a foil that could yield 10 μCi/min at 325°C would yield only about 4 μCi/min at 285°C.

The following example is included to show how this $^3H$ emanation relates to the AEC safety specifications. The State of California is referenced in this discussion since California has been authorized under the "agreement state" act by the Atomic Energy Commission:

> In the California Administrative Code, Title 17, the maximum permissible concentration (MPC) of $^3H$ in (room) air is $5 \times 10^{-6}$ μCi/ml. It is necessary to make two assumptions so that the release rate from the chromatograph can be related to this MPC. A laboratory volume of 2000 $ft^3$ will be assumed to be the smallest practical size laboratory room. The vast majority of laboratory rooms are considerably larger. The ventilation rate assumed for this calculation is 2 room volumes per hour. This is a very conservative figure based on the California building code. The $^3H$ emanation rate of 5 μCi/min will be used for calculation purposes although our previously reported data showed a maximum average $^3H$ emanation rate of 5.5 μCi/min for $Sc^3H$ at 325°C.
>
> The equilibrium condition described by
>
> $$N = \frac{R}{\lambda}(1-e^{-\lambda t})$$
>
> Where N = Concentration of radioactivity
> R = Emanation rate = 5 μCi/min
> λ = Exhaust rate = 66.6 $ft^3$/min
> t = Time
>
> is a commonly accepted method of calculating the concentration of radioactivity in a closes volume (the activity is assumed to be homogeneously distributed within the closed volume).

At equilibrium, the buildup factor, $1 - e^{-\lambda t}$ approaches unity and the equation reduces to $N = R/\lambda$.

Then:

$$N = \frac{5\ \mu Ci/min}{66.6\ ft^3/min} = 0.075\ \mu Ci/ft^3$$

$$= 2.6 \times 10^{-6}\ \mu Ci/ml$$

This is about half the MPC of $5 \times 10^{-6}$ μ Ci/ml.

Complete elimination of $^3H$ in the atmosphere may be accomplished by venting the cell to a fume hood or through an exterior window. A 10 foot length of PTFE tubing (1/16" ID Part No. 24-000472-00) has been provided for this purpose.

## 3.12 SUMMARY OF OPTIMUM CONDITION

The detectivity specifications stated in the introduction section referred to an optimized condition yielding 0.1 pg detection with a Lindane sample. The topics detailed in this section are summarized in brief as reminders of the procedures that must be followed if sub pg of Lindane is to be successfully detected.

1) Non absorptive chromatographic system (3.10).

2) "Ideal" system except for column (3.4).

3) New $Sc^3H$ foil or at least a reconditioned foil yielding greater than $2 \times 10^{-8}$ amp standing current (63% pen deflection x attenuation 32 x sensitivity $10^{-9}$).

4) Optional noise filter, 1 sec (3.2).

5) Accurate calibrated standard.

6) Moderate flow of carrier, 20-30 ml/min and no make-up (3.11).

7) Gas tight septum and syringe.

A chromatogram of 1 picogram Lindane is shown in Figure 3-9: an 8% peak is shown in the trace under standard conditions (-90 Vdc, 2 m sec noise filter).

FIGURE 3-9 1.0 picogram LINDANE

# SECTION 4
# GENERAL INSTALLATION

## 4.1 GENERAL

Installation of the $Sc^3H$ ECD is basically identical in Aerograph instruments with the exception of the 2100 series. Some installation steps that differ with various model instruments are detailed in Section 5. Installation steps common to all instruments are described in the following steps.

Detector temperature is monitored at the foil zone rather than detector base. The temperature differential is less than 10°C between the cell inlet and the foil. The pyrometer reads within 2°C of the true foil temperature. See Figure 4-1 showing tower detail.

The foil holder facilitates foil removal and reinstallation. Down time for foil removal is minimized since the operator need not cool down the detector base and tower, thus he is encouraged to remove the foil during extended periods of non-use thereby increasing its usable life. Restabilization of standing current following foil installation typically requires ~ 20 minutes. Sturdy ceramic insulators greatly reduce susceptibility to accidental damage. The extended cell exit tube provides a readily accessible point to connect to a fume hood vent line and simplifies hook-up to gas flow measuring devices. See Figure 4-2 or 4-3.

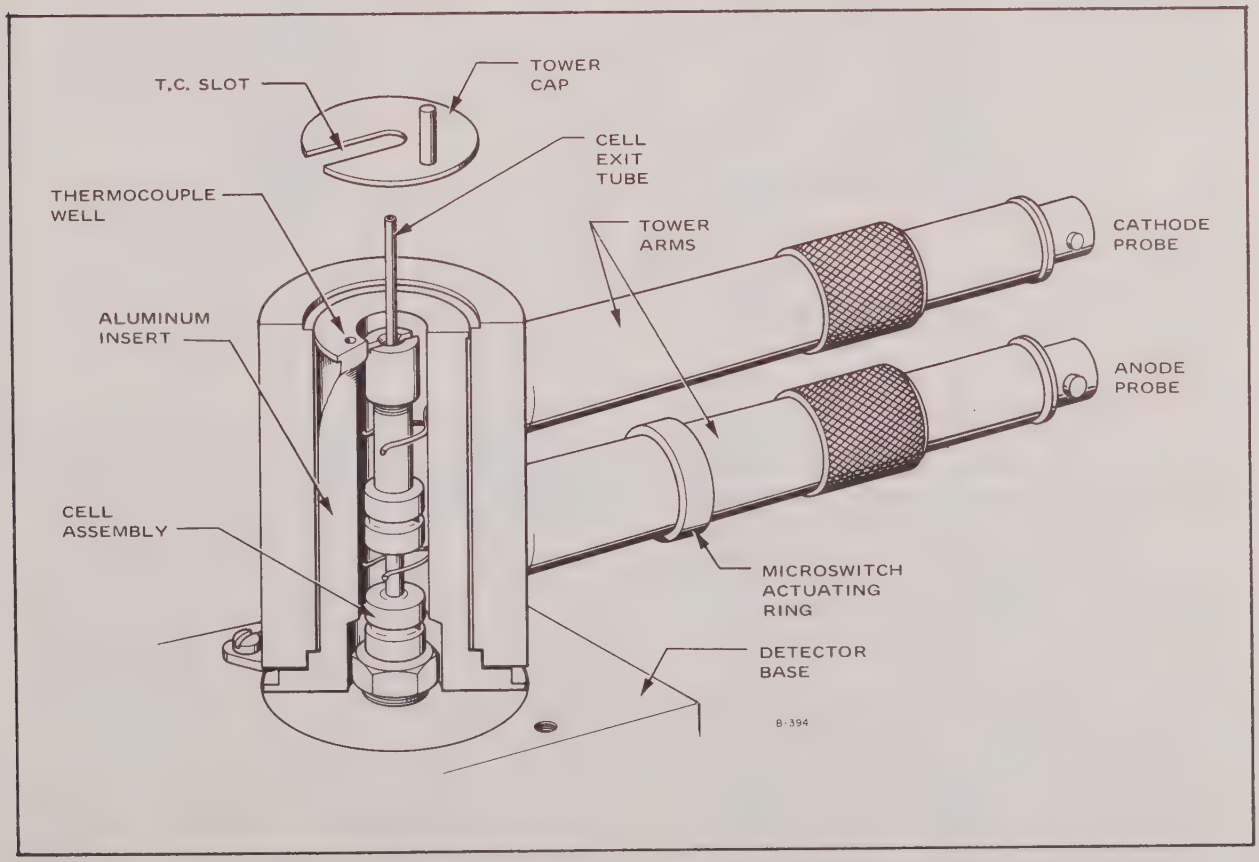



FIGURE 4-1 $Sc^3H$ ECD ASSEMBLY

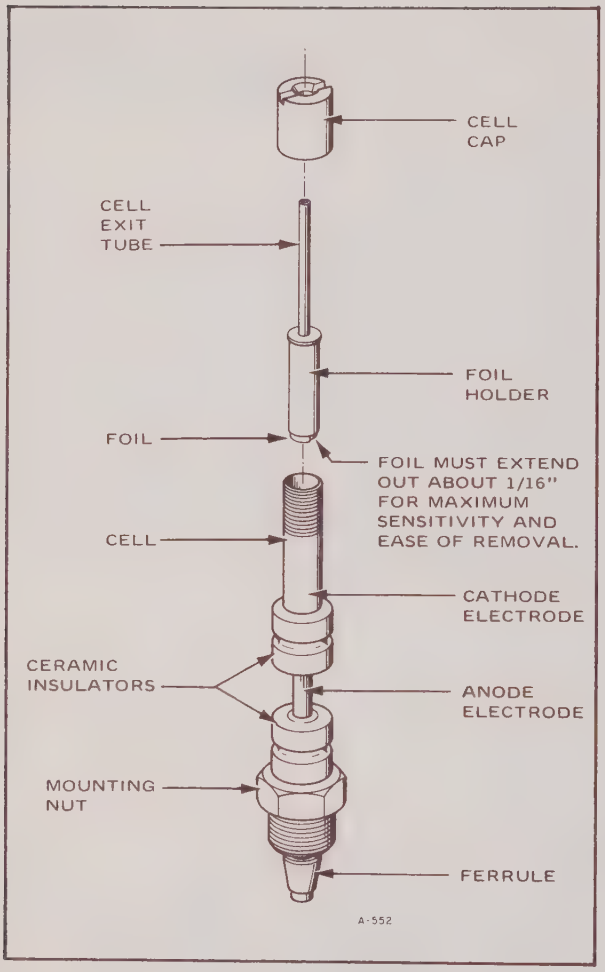



FIGURE 4-2 $Sc^3H$ ECD CELL ASSEMBLY

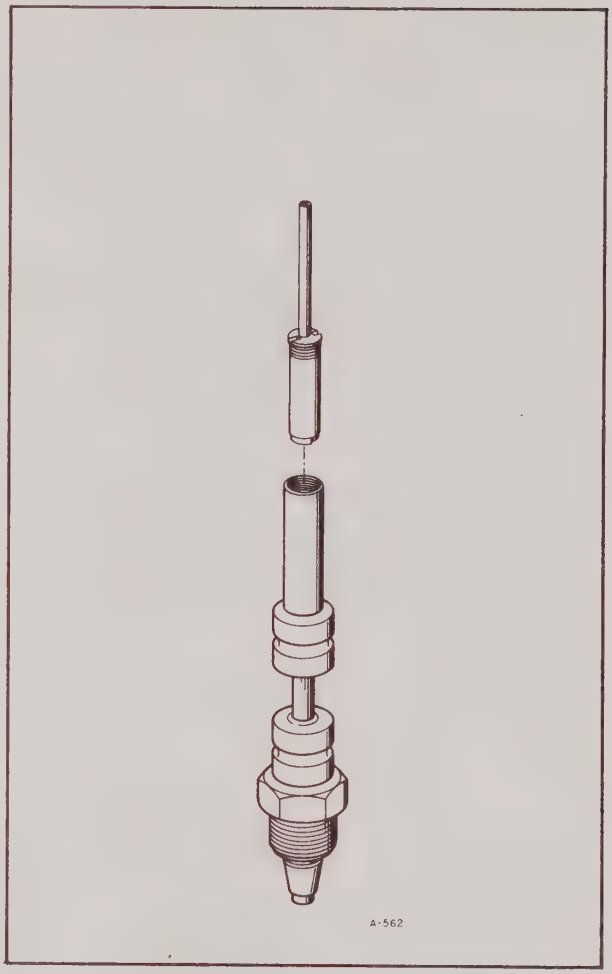



FIGURE 4-3 $Sc^3H$ ECD CELL ASSEMBLY (EARLIER DESIGN)

Every Aerograph instrument equipped with the $Sc^3H$ ECD is modified to limit the detector temperature to 325°C. The limiting switch (Fenwal) is activated by a microswitch that is in turn activated when a $Sc^3H$ ECD assembly is installed. The actuating plate of the microswitch is depressed by a ring (shown in Figure 4-1) on the lower electrode housing when the tower is secured into position.

When interchanging detector assemblies, it should be carefully determined that the switch is functioning; physical operation of the microswitch is indicated by an audible "click" when the actuating plate is depressed. Conventional detectors, i.e., FID, AFID, will not depress the switch plate since they do not have the ring on the lower electrode housing.

ECD operating procedures for instrument series 1400, 1700, 1800, 2100, 2400, 2700, and 2800 are described in the following text. It is strongly recommended that prior to actual operation of the $Sc^3H$ ECD, the operator have a good understanding of detector principle and characteristics, as described in Section 3. Selection of optimum operating parameters are dictated primarily by sample composition and specific data desired, however, the following guidelines are offered for general routine operation.

Standard precautions and regulations that must be observed by the users of radioactive sources are described in the AEC pamphlet, AEC Rules and Regulations, Title 10, Chapter 1, Part 20. State licensees will receive the appropriate pamphlets with regulations and precautions from the State.

## 4.2 DETECTOR CELL AND TOWER ASSEMBLY INSTALLATION

**CAUTION**

**Every instrument incorporating a $Sc^3H$ ECD must display AEC approved warning labels. Each detector kit contains two labels; one affixed permanently to the detector tower assembly displaying the official magenta and yellow radioactive symbol and an adhesive backed label (VA Part No. 31-000322-00) reading "WARNING—this instrument not authorized for $Ti^3H$ detectors", which must be conspicuously displayed on the instrument.**

1) It is most important to maintain oil and dust free surfaces of ceramic insulators. Avoid hand contact with ceramic insulators when working with the cell assembly.

2) With the 7/16" socket wrench provided, secure the $Sc^3H$ EC detector cell assembly without foil, foil holder, and cell cap firmly into the detector base (nut and ferrule are pre-swaged at the factory).

3) It is most important that good contact be maintained between tower and detector base surface. Be sure both surfaces are free of dust, shreds of insulation or similar particulates.

4) Carefully lower the detector tower over the cell assembly.

5) Secure the tower in place by guiding locking tabs into the groove with clockwise rotation at the base of the tower. Tighten mounting screws, retighten after heating to insure maximum contact.

6) Install electrodes using horizontal alignment keys and slots to properly orient electrodes in tower electrode housing. Upper (cathode) polarizing electrode is identified by a 2 pin BNC cable connector, and the voltage selector screw which should be installed upward. The lower (anode) polarizing electrode has a single pin BNC cable connector. Carefully guide the electrodes into tower arm until probe clips engage the detector cell assembly. At the end of insertion motion, a slight "snap" action should be felt.

7) Check final installation to be sure cell assembly is centered and does not contact tower surfaces which would short out cell voltage.

8) Connect cables. Cables cannot be interchanged due to pin configuration. Turn on cell voltage.

**WARNING**

**With cables connected and electrometer cell voltage ON, a -90 Vdc potential is present on the cell assembly. Disconnect cables and turn electrometer cell voltage OFF before working on detector tower assembly.**

9) After electrodes have been secured, a VOM check should be made between the cell exit tube and chassis ground (meter should read ~-90 Vdc. VOM input impedance will result in a slightly lower reading). If no reading, withdraw the electrode (cathode), realign the clip and reinsert.

   Check the lower (anode) electrode for proper contact with the cell; insert a paper clip into the center pin receptacle of the connector. With the VOM, set on "Ohms Resistance" check for continuity between the paper clip and tower body. Reading should be infinity. A continuity reading indicates shorting between electrode and tower. Withdraw the electrode, align the clip and reinsert.

10) Install foil and holder. Secure cell cap with special screwdriver. Recheck to be sure cell is centered in the tower.

11) Install tower cap with slot positioned to expose thermocouple wells in the aluminum insert (specific instructions for thermocouple placement are covered in Section 6).

**NOTE**

**With temperature switch set to Detector, pyrometer reads approximate foil temperature. If instrument is converted to FID or AFID applications, standard thermocouple arrangement should be established.**

12) Included in each $Sc^3H$ ECD kit is a 12" x 12" pad of insulation to be added after the installation of the $Sc^3H$ ECD. This added insulation is required to reduce temperature gradients due to heat loss through the tower. Installation of additional insulation varies with each instrument, therefore detailed insulation instructions are discussed in Section 6 under the specific instrument.

13) Turn on heat.

# SECTION 5
# INDIVIDUAL INSTRUMENT INSTALLATION

## 5.1 GENERAL

In addition to ECD exchange and installation instructions common to all Aerograph GC instruments covered by this manual, individual instruments require installation steps unique to themselves. Such steps pertinent to specific models are detailed in the following paragraphs.

## 5.2 SERIES 1400 and 2400 INSTRUMENTS

Series 1400 and 2400 instruments require Kit No. 96-000093-00. Table 5-1 lists the parts and part numbers of the kit contents. Check the list against contents to be sure all parts listed are received.

TABLE 5-1

| Description | Part No. | Quantity |
|---|---|---|
| S/A Tower | 01-000920-01 | 1 ea |
| Cap, detector | 13-000060-00 | 1 ea |
| Assembly, probe, anode (bottom) | 02-000954-01 | 1 ea |
| Assembly, cell, $Sc^3H$ | 02-001681-01 | 1 ea |
| Assembly, probe, cathode (top) | 02-001682-02 | 1 ea |
| Tubing, Teflon, 1/16" OD | 37-000416-00 | 10 ft |
| Label, caution, "Radioactive material" (affixed to vial) | 31-000347-00 | 1 ea |
| Vial, foil container | 66-000118-00 | 1 ea |
| Brkt, Label Mtg., (riveted to tower) | 04-000924-00 | 1 ea |
| Label, radioactive, (affixed to tower) | 31-000320-00 | 1 ea |
| Label, warning, "Radioactive material" | 31-000322-00 | 1 ea |
| Screwdriver | 72-000065-00 | 1 ea |
| Thermocouple | 52-000062-00 | 1 ea |
| Wrench, 7/16", spintight | 72-000041-00 | 1 ea |
| Manual, $Sc^3H$ ECD | 85-001118-17 | 1 ea |
| Insulation | 24-000703-00 | 1 $ft^2$ |
| Stud, drive groove | 15-005002-00 | 2 ea |
| Ferrule, graphite, 1/4" | 16-000716-00 | 2 ea |
| Ferrule, back brass, 1/4" | 16-000044-00 | 2 ea |
| Tubing, Teflon, 1/16 ID | 24-000439-00 | 3 in |
| Snap bushing | 15-000989-00 | 1 ea |

Test "click" sound of microswitch operation by manually depressing microswitch or microswitch plate before tower installation.

Figures 5-1 and 5-2 show exploded views illustrating the order of installation in the 1400 and 2400 instruments. To install the thermocouple (VA Part No. 52-000062-00), insert the fused junction into the well of the tower as far as it will go. Replace the tower cap.

Additional insulation must be installed over the detector towers and around tower arms. Cut sections of insulation as per applicable template on the last page of the manual. Add small pads around tower arms where required. Ample insulation is included for future replacements.

**NOTE**

**Right side panel must be punched as per Figures 5-1 and 5-2 to accommodate the extra long multi-voltage cathode probe on 2400 B channel installation.**

The foil temperature will be indicated when the pyrometer selector is set to the DETECTOR position.

Limited head space between oven lid and cell exit tube on 2400 instruments require that the exit tube be bent to accommodate a Teflon vent line. Make a right angle bend (1/4" to 3/8" radius bend) in the tube perpendicular to the cell cap screwdriver slot about mid distance of the tube length. Tube may be bent with fingers or needle nose pliers. Check to be sure flow has not been impeded.

The above instructions are not applicable where hole is already provided in the oven lid.

## 5.3 SERIES 1700/1800 and 2700/2800 (Moduline)

Installation of the ECD on Moduline instruments require Kit No. 96-000095-00. Check the list in

Table 5-2 against the parts received to be sure all components have been shipped.

TABLE 5-2

| Description | Part No. | Quantity |
|---|---|---|
| S/A Cover Box | 01-000693-00 | 1 ea |
| S/A Tower | 01-000920-01 | 1 ea |
| Cap, detector | 13-000060-00 | 1 ea |
| Assembly, probe, anode (bottom) | 02-000954-01 | 1 ea |
| Assembly Cell, $Sc^3H$ | 02-001681-01 | 1 ea |
| Assembly, probe, cathode (top) | 02-001682-02 | 1 ea |
| Tubing, Teflon, 1/16" OD | 37-000416-00 | 10 ft |
| Label, caution, "Radioactive material" (affixed to vial) | 31-000347-00 | 1 ea |
| Brkt, label mtg., (riveted to tower) | 04-000903-00 | 1 ea |
| Label, radioactive, (affixed to tower) | 31-000320-00 | 1 ea |
| Label, warning, "radioactive material" | 31-000322-00 | 1 ea |
| Insulation | 24-000703-00 | 1 $ft^2$ |
| Wrench, 7/16" spintight | 72-000041-00 | 1 ea |
| Manual, $Sc^3H$ ECD | 85-001118-17 | 1 ea |
| Tubing, Teflon, 1/16" ID | 24-000439-00 | 3 in |
| Vial, foil container | 66-000118-00 | 1 ea |
| Screwdriver | 72-000065-00 | 1 ea |
| Ferrule, graphite ¼" | 16-000716-00 | 2 ea |
| Ferrule, back brass ¼" | 16-000044-00 | 2 ea |

1) Remove detector oven top cover, insulation pad, and inner aluminum plate. After system and column preconditioning, install ECD cell and tower as shown in Figure 5-3.

2) Cut circular pads of insulation to close open hole in the insulated tower cover.

3) Replace detector oven covers and insulation.

4) Read foil zone temperature on the DETECTOR position of the pyrometer selector switch.

## 5.4 2100 SERIES

Series 2100 instruments require ECD Accessory Kit No. 96-000094-00. Check parts listed in Table 5-3 with parts received to be sure all components have been shipped.

TABLE 5-3

| Description | Part No. | Quantity |
|---|---|---|
| Insulation, detector tower | 24-000166-00 | 1 ea |
| S/A Tower | 01-000920-01 | 1 ea |
| Insulation | 24-000703-00 | 1 $ft^2$ |
| Cap, detector | 13-000060-00 | 1 ea |
| Assembly, probe, anode (bottom) | 02-000954-01 | 1 ea |
| Assembly, cell, $Sc^3H$ | 02-000 | 1 ea |
| Assembly, probe, cathode (top) | 02-001682-02 | 1 ea |
| Thermocouple | 52-000062-00 | 1 ea |
| Adapter, heat conductor | 16-000751-00 | 1 ea |
| Vial, foil container | 66-000118-00 | 1 ea |
| Brkt., Label Mtg., (riveted to tower) | 04-000924-00 | 1 ea |
| Label, radioactive, (affixed to tower) | 31-000320-00 | 1 ea |
| Label, warning, "radioactive material" | 31-000322-00 | 1 ea |
| Label, caution, "radioactive material" (affixed to vial) | 31-000347-00 | 1 ea |
| Tubing, Teflon, 1/16" OD | 37-000416-00 | 10 ft |
| Wrench, 7/16" spintight | 72-000041-00 | 1 ea |
| Manual, $Sc^3H$ ECD | 85-001118-17 | 1 ea |
| Screwdriver | 72-000065-00 | 1 ea |
| Clamp, tower | 08-000072-00 | 2 ea |
| Screw, 8-32 x 7/16" PHL PHD | 12-222008-07 | 2 ea |
| Stud, drive groove | 15-005002-00 | 2 ea |
| Ferrule, graphite ¼" | 16-000716-00 | 2 ea |
| Ferrule, back brass ¼" | 16-000044-00 | 2 ea |
| Tubing, Teflon, 1/16" ID | 24-000439-00 | 3 in |

The detector base on 2100 instruments requires an additional aluminum heat conducting adapter (base collar) for mounting the $Sc^3H$ ECD assembly.

1) Replace the existing ring adapter with the base collar included in the kit. (see Figure 5-4)

2) Install the $Sc^3H$ cell and tower assembly (without foil) after system and column have been thoroughly preconditioned.

3) Cut small circular pad of insulation to fill in the tower insulating pillow and reduce heat losses via top of tower. See Figure 5-4.

4) Read foil zone temperature on the DETECTOR position of the pyrometer selector switch.

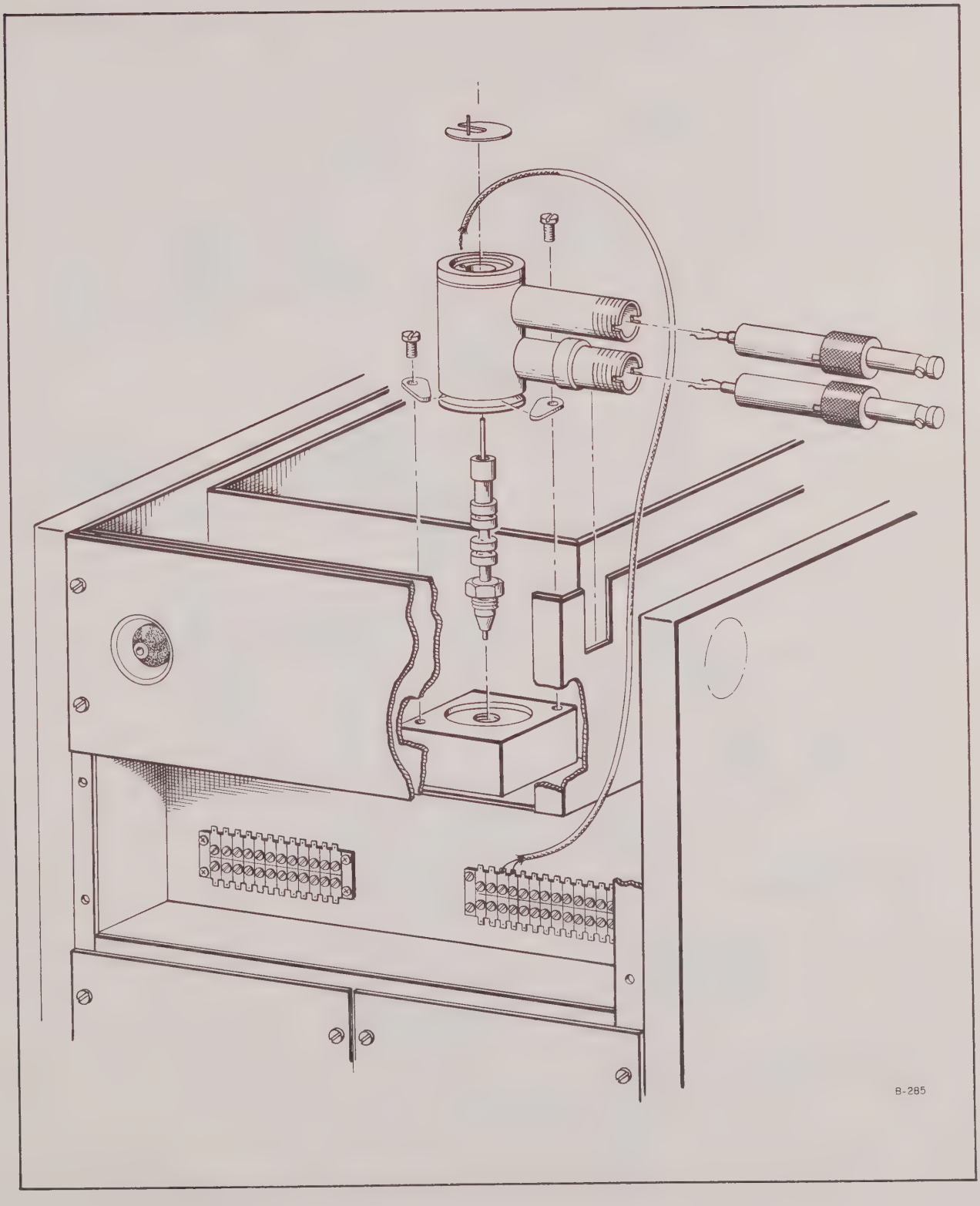



FIGURE 5-1 $Sc^3H$ ECD INSTALLATION (1400)

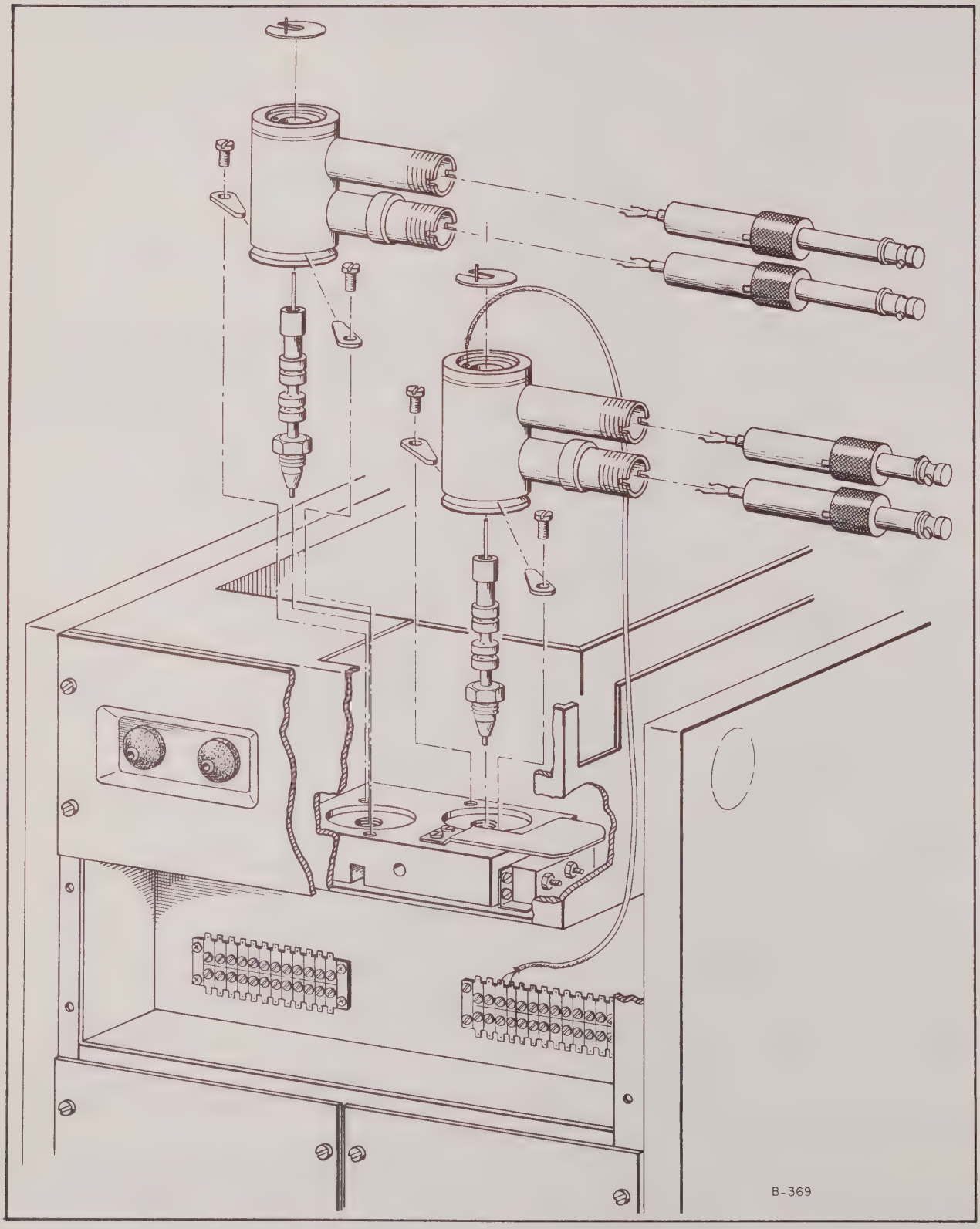



FIGURE 5-2 $Sc^3H$ ECD INSTALLATION (2400)

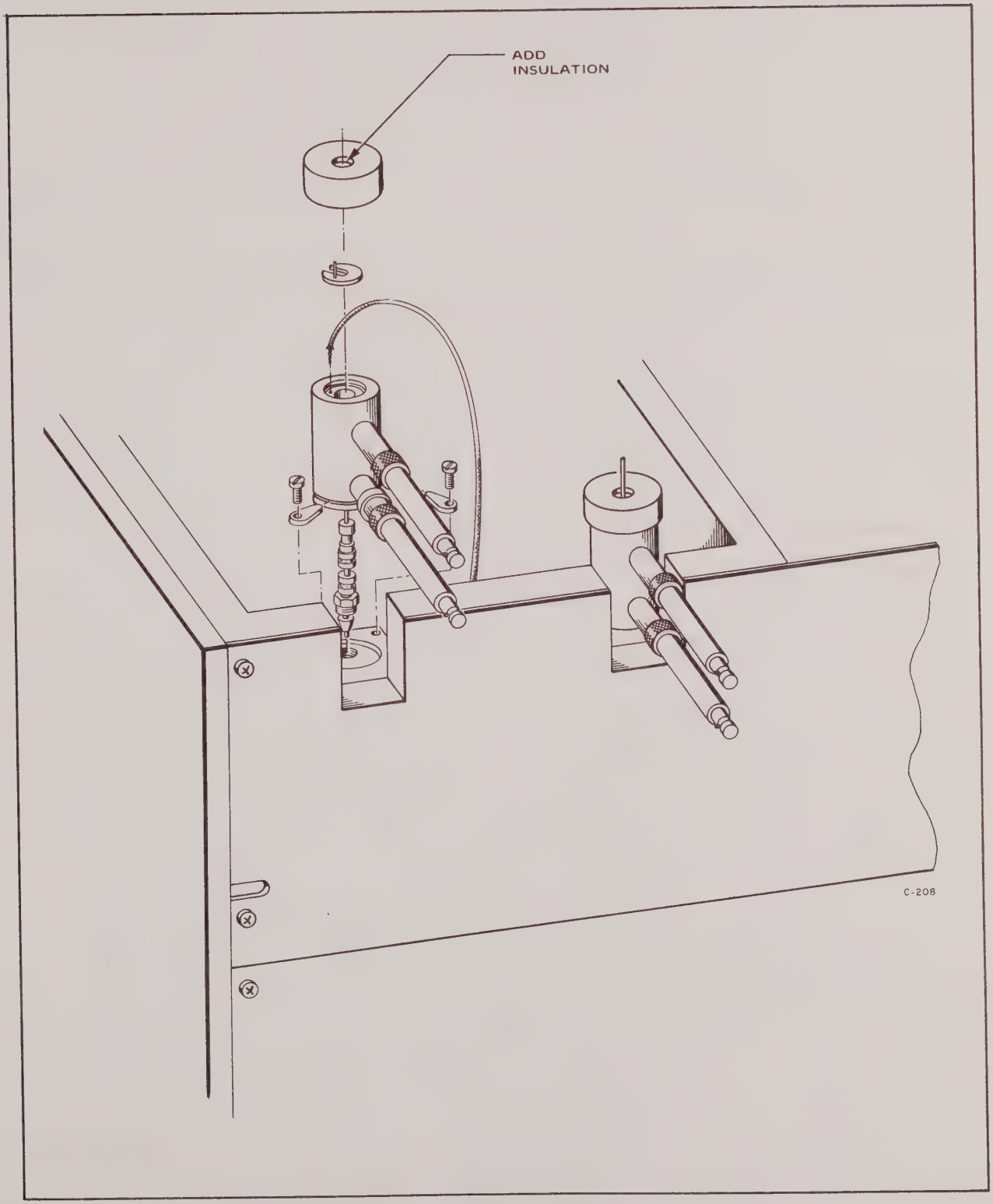



FIGURE 5-3 $Sc^3H$ ECD INSTALLATION (1700/1800/2700/2800)

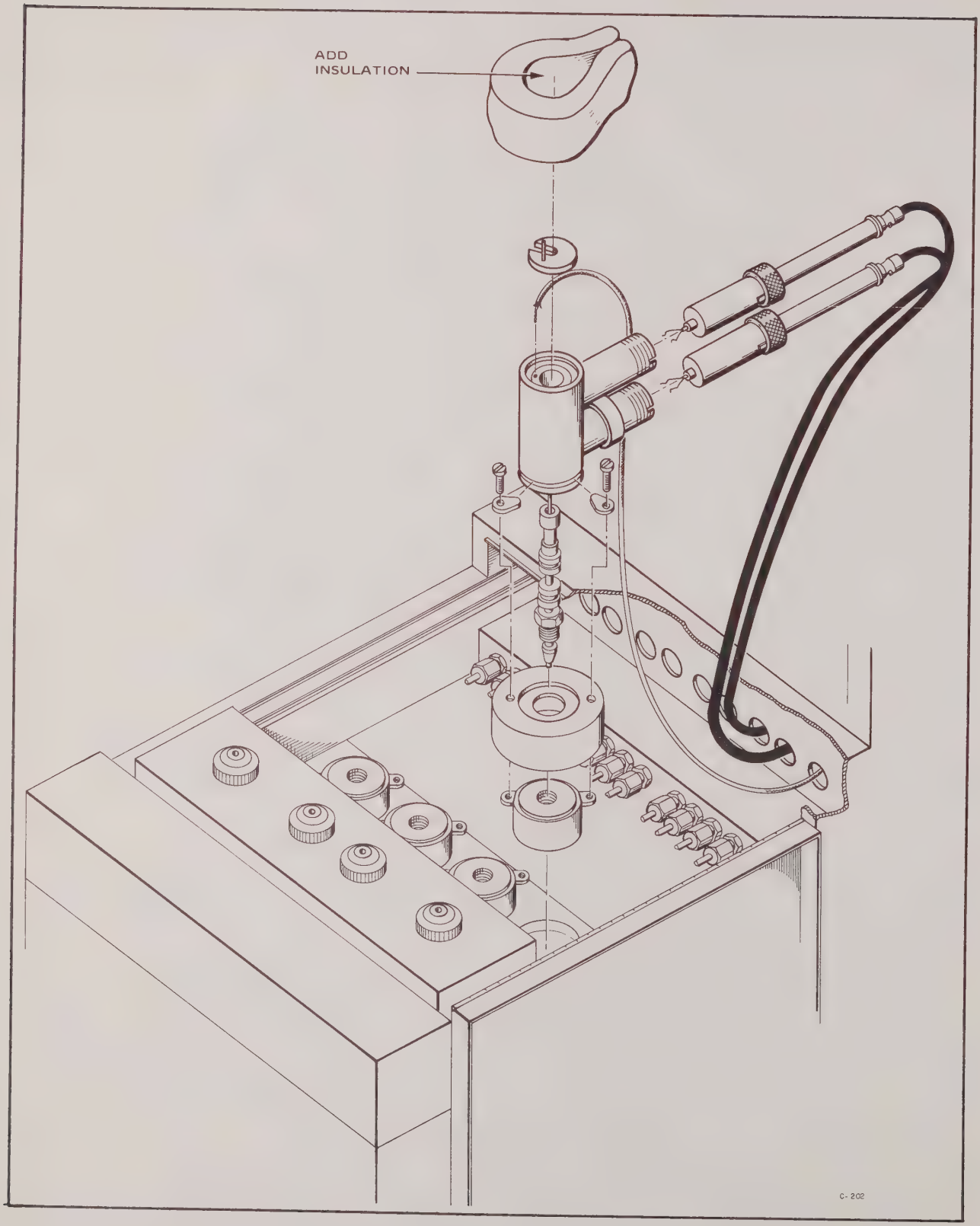



FIGURE 5-4 Sc$^{3}$H ECD INSTALLATION (2100)

# SECTION 6
# OPERATION

## 6.1 PREINSTALLATION PREPARATION AND OPERATION

This section briefly summarizes the recommended procedures for preconditioning system and column, and typical instrument control settings preparatory to performing an ECD GC analysis.

### 6.1.1 Carrier Gas

- High purity nitrogen is preferred (<5 ppm oxygen and water vapor). For maximum protection, installation of Oxygen Trap (VA Part No. 68-000056-00) in the carrier gas line is recommended. Trap capacity is sufficient to protect against one full cylinder of contaminated $N_2$. Trap replacement with each new cylinder of gas is recommended.

**CAUTION**

**Hydrogen may be used as carrier gas for rare applications, but MUST NOT be used above 100°C.**

- Where $Sc^3H$ detectors only are installed, the HYDROGEN inlet fitting must be capped off to prevent splitting the flow. With instruments employing dual detectors, shut off the HYDROGEN metering valve to the ECD, or attach separate $N_2$ supply for make-up gas.

**NOTE**

**Metering valves are easily damaged by excessive tightening. Tighten only enough to effect shut off of flow.**

### 6.1.2 Column Conditioning

Prior to installation of the $Sc^3H$ ECD, the selected column should be preconditioned to minimize column bleed during operation.

1) Remove existing tower and detector cell assembly.

2) Cap off the injector port with a blank off nut, or aluminum foil backing on the septum.

3) Install the column. Use graphite ferrules for glass column installation, as outlined in Section 9.

4) Set detector base temperature at about 25-50°C above recommended column conditioning temperature for applicable stationary phase.

5) When conditioning metal columns, it is recommended that the exit end of the column *not* be connected to the detector base fitting, however, glass columns should be connected at both ends to prevent strain.

6) Set the carrier gas flow to ~30 ml/min.

7) Conditioning temperature is dictated by the maximum allowable operating temperature of the stationary phase of the column packing. Table 6-1 lists several of the more commonly used stationary phases. Conditioning temperatures are 25°C lower than listed maximum operating temperatures. Refer to the Aerograph Chromatography Catalog for a comprehensive list of stationary phase specifications.

8) Condition the column for a minimum of 16 (overnight) hours. Work schedule permitting, up to 64 (weekend) hours of conditioning is recommended.

TABLE 6-1
COLUMN-CONDITIONING TEMPERATURES

| Stationary Phase | Conditioning Temperature/°C |
|---|---|
| DC-200 | 225 |
| OV-1 | 325 |
| OV-17 | 275 |
| OV-101 | 325 |
| OV-210 | 250 |
| OV-225 | 250 |
| QF-1 | 225 |
| SE-30 (GC Grade) | 275 |
| SE-52 | 275 |

### 6.1.3 Instrument Decontamination (Instruments in previous service)

Experience has shown that injector and detector systems operated at recommended temperatures are not significantly contaminated by condensation deposition, however, if your particular applications result in appreciable contamination, the following procedure for steam cleaning is suggested.

1) Remove detector assembly.

2) Install a length of empty tubing between injector and detector fittings and set carrier gas at 30 ml/min.

3) With a conditioned septum in the injector, raise oven, injector and detector temperature to 300°C and make 5 successive injections of 100 $\mu$l each of distilled water.

### 6.1.4 Standing Current Check

The standing current should be checked when the detector is initially installed and periodically thereafter. The standing current value should remain relatively constant after initial equilibration. A current increase is indicative of foil equilibration which will usually stabilize within 16 hours, and a gradual decrease in standing current usually indicates foil contamination.

With stable temperatures and carrier gas flow, check standing current by the following procedure:

1) Electrometer Settings

   Set RANGE switch to $10^{-9}$ and attenuator at 32.

   For single channel electrometers, recorder polarity is automatically reversed when switch is in the EC or OFF position.

   For ECD operation in channel A, recorder leads reversal is necessary.

2) Adjust BUCKING control to position pen on scale.

3) Set CELL VOLTAGE switch to OFF.

4) Attenuator to 64 if necessary.

5) Read recorder pen deflection—convert to standing current.

   Example: If recorder pen reads 70% of full scale, attenuator at 32 and RANGE at $10^{-9}$ amp.

   Calculate: $0.70 \times 32 \times 10^{-9} = 22.4 \times 10^{-9}$
   amps $= 2.24 \times 10^{-8}$

6) Standing current should be $\geq 2 \times 10^{-8}$ amps for new foil, "ideal" system, and well conditioned columns.

### 6.1.5 Instrument and Electrometer Control Settings

**WARNING**

**2100 Series Instruments—The DETECTOR SAFETY LIMIT switch MUST be set to ECD SCANDIUM position or the ON position of earlier instruments..**

1) Set the detector temperature control (typically 50°C higher than maximum column oven temperature). Allow the detector temperature to equilibrate at the control setting.

2) Set column oven temperature controls for isothermal operation. (ECD's cannot effectively handle the changing column bleed in temperature programmed columns).

3) Set injector temperature.

4) Turn off carrier gas and install foil holder. Allow 5 minutes for foil to heat.

5) Set carrier gas valves for desired flow rate flow measurement setup.

6) Set Electrometer Controls

   Set RANGE switch to $10^{-9}$ (most widely used sensitivity) or $4 \times 10^{-10}$ for those interested in maximum useable sensitivity.

   —Single Channel, set CELL VOLTAGE to EC.

   —Dual Channel, set MODE switch on FL/EC for B channel. For ECD operation in channel A, manual reversal of recorder leads is required.

7) Check $Sc^3H$ standing current as described in paragraph 6.2.4.

8) Loosen voltage select screw about two turns counterclockwise on polarization electrode to give -90 Vdc.

9) Set electrometer CELL VOLTAGE switch to EC position.

10) Set electrometer RANGE and ATTENUATOR switches for required sensitivity.

11) Adjust electrometer BUCKING control for electrical zero setting of recorder pen.

**6.1.6 Foil Removal**

Foil life is significantly increased if foil is removed from the heated detector zone when not in use (overnight, over week-ends, holidays, etc.) The foil holder design facilitates easy foil removal without the need to remove the cell assembly. A 12-15 minute reequilibration time is typical. See Figure 4-2 showing details of foil holder.

**WARNING**

**All detector assembly parts are HOT. Use long nose pliers or other suitable tool for handling. Radioactive foil must never be touched by hand. Tools used for handling the foil must be washed after each use.**

1) Turn off EC voltage.

2) Where applicable, disconnect plastic vent line.

3) Remove thermal insulation.

4) Remove detector tower cap.

5) With special screwdriver provided (VA Part No. 72-000065-00), unscrew cell cap.

6) Grasp cell exit tube and withdraw cell cap, foil holder, and foil (cap remains with foil and holder when stored).

7) Store foil holder assembly, complete with cap in the suitably labeled container provided. Store the container so as to protect against accidental misplacement or loss.

# SECTION 7
# TROUBLESHOOTING

## 7.1 GENERAL

Table 7-1 lists some of the problems that may be encountered with the $Sc^3H$ ECD. If the detector appears faulty, a possible cause and remedy may be listed in Table 7-1. If the problem cannot be corrected, contact an Aerograph Sales or Service Representative.

TABLE 7-1
$Sc^3H$ ECD OPERATOR TROUBLESHOOTING CHART

| Symptom | Possible Cause | Check and/or Remedy |
|---|---|---|
| No standing current. | Signal or cell voltage cables not making contact or open. | Ensure detector electrodes and cables are installed properly. |
| | Cell voltage shorted to ground. | Ensure detector exit tube (cell assembly cap) is not touching the tower cap. |
| | Septum too tight, obstructing carrier gas flow. | Tighten injector nut just enough to prevent leaks. |
| | Hydrogen supply not disconnected and capped off from instrument. | Hydrogen and/or air leaking into the chromatographic system will quench the standing current. |
| Low standing current. | Leak in chromatographic system. | Pressure check the chromatographic system. |
| | Detector electrodes improperly installed. | Ensure electrode clip leads make firm contact to cell. |
| | Contaminants in the carrier gas. | Use only high-purity nitrogen (oxygen trap highly recommended); steam clean GC system. |
| | Contaminants in the sample. | Clean the sample (i.e., extract lipids, pigments, etc.) |
| | Septum too tight, obstructing carrier gas flow. | Tighten injector nut just enough to prevent leaks. |
| | Hydrogen supply not disconnected and capped off from instrument. | Hydrogen and/or air leaking into the chromatographic system will quench the standing current. |
| Normal standing current—low sensitivity. | Sample not passing through column in tact. | Repack 6" of column packing on injector end. Or, repack entire column. |
| High standing current—low sensitivity. | Major portion of observed standing current is leakage current. | Remove foil, check standing current (clean ceramic insulators with acetone or a very fine abrasive). |

TABLE 7-1 Sc$^3$H ECD OPERATOR TROUBLESHOOTING CHART (cont.)

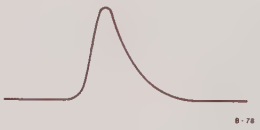
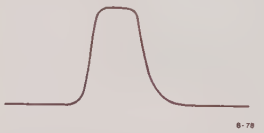
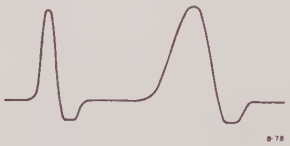
| Symptom | Possible Cause | Check and/or Remedy |
|---|---|---|
| Unstable baseline | Leak in chromatographic system. | Leak check chromatographic system. |
| | ECD just installed. | Allow 16 hours to equilibrate. |
| | Detector temperature not equilibrated. | Allow detector temperature to stabilize. |
| | Abrupt changes in environmental temperature. | Ensure that detector is not located near an air conditioning vent, etc. Cover exposed portion of detector with insulation. |
| | Contaminated chromatographic system. Bad septum. | Clean the chromatographic system and detector as described in manual. Replace septum and/or add extender. |
| Tailing peaks.  | Column-oven temperature too low. | Increase column-oven temperature. |
| | Poor sample injection technique. | Refer to syringe manufacturer's instruction sheet. |
| | Sample interacting with liquid phase or solid support. | Ensure correct column has been selected for the type of analysis. |
| | Carrier gas flow rate too slow. | Increase carrier gas flow rate. |
| | Leak in chromatographic system. | Locate and correct leak. |
| Rounded-off peaks.  | Detector overloaded and operating out of detector's linear range. | Reduce sample size and/or dilute sample. |
| Negative dip after peaks.  | Contaminated detector foil. | Clean detector as described in manual. Increase detector temperature if possible. |
| Low sensitivity with normal retention time. | Insufficient sample size. | Increase sample size. |
| | Attenuation too high. | Decrease electrometer ATTENUATOR setting. |
| | Leak in chromatographic system. | Locate and correct leak. |
| | Contaminated detector. | Clean detector as described in manual. |
| | Contaminated column. | Replace or recondition column. |
| | Contaminated carrier gas. | Replace oxygen trap. Ensure high-purity nitrogen is being used. |
| | Decomposition of sample. | Ensure correct column, temperatures, and apparatus have been selected for the type of analysis. |

TABLE 7-1 Sc$^3$H ECD OPERATOR TROUBLESHOOTING CHART (cont.)

| Symptom | Possible Cause | Check and/or Remedy |
|---|---|---|
| Low sensitivity with long retention time. | Leak in chromatographic system. | Locate and correct leak (probably at injector) |
| | Carrier gas flow rate too low. | Increase carrier gas flow rate. |
| | Carrier gas flow system obstructed. | Check for foreign materials in gas flow system. Ensure septum is not obstructing carrier gas flow. |
| Sawtooth baseline. | Defective diaphragm in carrier gas flow controller. | Replace diaphragm or flow controller. |
| Baseline drifting upscale. | Standing current decreasing because of column bleed. | Replace or recondition column. |
| | Column, injector, or detector temperatures changing. | Ensure column, injector and detector temperatures have stabilized. |
| Baseline drifting downscale. | Standing current increasing (good sign). | Normal. Standing current usually increases for several hours after installation. |
| Sinusoidal baseline. | COLUMN SAFETY LIMIT control being used as temperature controller. | Use only as upper limit cut off. Use column-oven controller to set the column-oven temperature. |
| | Detector temperature cycling. | Faulty detector temperature controller. Replace temperature controller. |
| | Column-oven door not closed properly. | Ensure column-oven door seals when closed. |
| | Faulty column-oven temperature controller (especially with high-bleed columns). | Replace column-oven temperature controller. |
| | Liquid phase bleed; Series 1400, 2100 and 2400 Instruments. | Remove approximately one inch of column packing material from detector end of column. |

# SECTION 8
# ROUTINE MAINTENANCE

## 8.1 GENERAL

When loss of sensitivity or an abnormal standing current develops, cleaning of the $Sc^3H$ foil may be required. This is the only maintenance needed except complete replacement of cell or foil due to physical damage or exhausted foil. The following cleaning procedure is for foil only. Do not subject the cell assembly with ceramic insulators to the same treatment. If cell contamination is determined to be the cause of faulty operation, we recommend that the cell be returned to the factory for cleaning.

## 8.2 FOIL REMOVAL

1) Remove the tower cover.

2) Disconnect exhaust tubing from cell exit tube (if one is installed).

3) Completely loosen cell cap with screwdriver (VA Part No. 72-000065-00).

4) With long nose pliers, lift out foil holder assembly complete with cap.

5) Extract the foil from the holder. Transfer the foil directly from the holder to the wash vial.

**WARNING**

**Always use gloves and tweezers when handling foil. Wash hands and all tools thoroughly after every such handling.**

## 8.3 FOIL CLEANING

1) Prepare 50 ml of 5% KOH in methanol (2.5 g pelleted KOH in 50 ml CP methanol).

2) Totally immerse the foil in ~15 ml of 5% KOH/Methanol solution in a tightly capped vial.

3) Place the vial in an ultrasonic cleaning apparatus containing ~1/2 inch of water; wash for one hour.

4) With clean tweezers, remove foil from vial and rinse thoroughly with 30-50 ml CP methanol. Allow foil to dry.

5) Discard wash residues to radioactive waste in accordance with AEC Regulations.

6) Insert the foil into the foil holder insuring a tight spring fit. Foil should protrude about 1/16" out of foil holder (refer to Figure 4-3).

7) Customers who prefer factory foil cleaning may ship the foil to Varian Aerograph, Walnut Creek, California. Foil must be shipped in a radioactive labeled container (recommend using labeled storage vial provided). Labeled containers must be packed in an outer shipping container. The outer container does not require a radioactive label, however, the shipping papers must stipulate 1 Ci $^3H$ gas adsorbed in a solid followed by the statement "no label required".

# SECTION 9
# GRAPHITE FERRULE INSTALLATION

## 9.1 GENERAL

Graphite ferrules have the advantage of being usable at higher temperatures than the Teflon® ferrules commonly used for installation of glass chromatographic columns. Ferrules may be reused in many cases if care is used in removal. Since graphite ferrules do not flow at elevated temperatures or "cold flow" at ambient temperatures, repeated tightening of fittings is not required.

## 9.2 INSTALLATION

For proper installation of the graphite ferrules using a standard metal Swagelok® back ferrule and metal nut, refer to Figure 9-1.

1) Install the nut as shown.

2) Install a reversed Swagelok metal backing ferrule to provide a flat back to back contact with the graphite ferrule.

3) Install the graphite ferrule with flat side toward nut and backing ferrule.

4) Insert column into the appropriate fitting, as far as possible, then retract just slightly so the glass column does not butt against the metal shoulder of the fitting.

5) First tighten the nut finger tight; then tighten an additional 1/4 turn with a small wrench.

## 9.3 REMOVAL AND REUSE

In many cases, the graphite ferrule may be removed from the column without damage in which case the ferrule will retain its sealing capabilities and can be reused.

For best results, remove the nut, back ferrule and graphite ferrule as a unit. Attempts to remove a used graphite ferrule separately can result in disintegration of the ferrule.

## 9.4 ORDERING INFORMATION

Table 9-1 lists the graphite ferrules available and Varian part numbers for ordering purposes.

TABLE 9-1

| Part Number* | |
|---|---|
| 02-001780-00 | 1/16" Graphite Ferrule |
| 02-001781-00 | 1/8" Graphite Ferrule |
| 02-001782-00 | 1/4" Graphite Ferrule |

*Part number includes appropriate metal back ferrule required for installation.

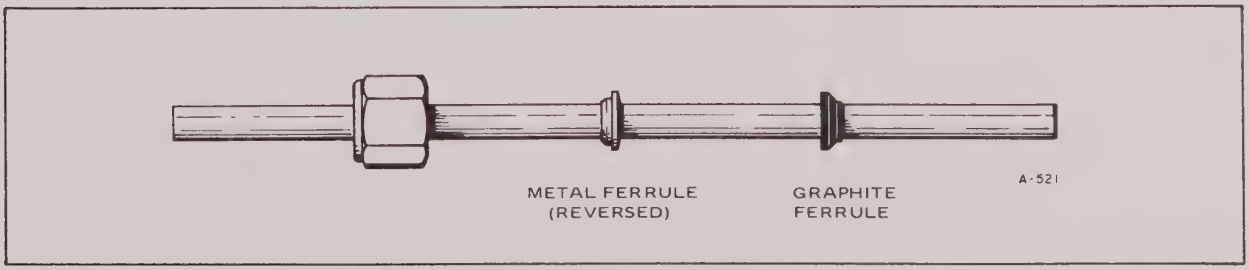



FIGURE 9-1 INSTALLATION OF GRAPHITE FERRULES

# SECTION 10
# ILLUSTRATED PARTS LIST

## 10.1 GENERAL

This section of the manual comprises parts identification drawings and parts lists.

The parts are identified on the drawings by an item number within a circle and listed numerically in the parts list. The numbers are followed by a corresponding part number and material description. Use the ten-digit part number and material description when ordering replacement parts. When ordering parts, also include the complete Model and Serial numbers of your instrument.

Listed below are the part numbers for components shown in Figure 10-1.

| Item No. | VA Part No. | Description |
|---|---|---|
| 1 | 13-000060-00 | Cap, Detector |
| 2 | 02-001682-02 | Assembly, Probe Cathode (top) |
| 3 | 02-000954-01 | Assembly, Probe, Anode (bottom) |
| 4 | 01-000920-01 | S/A Tower |
| 5 | 12-222008-07 | Screw, 8-32 x 7/16" Phl Phd |
| 6 | 08-000072-00 | Clamp, Tower |
| 7 | 02-001681-01 | Assembly, Cell, $Sc^3H$ |
| 8 | 16-000751-00 | Adapter, heat conductor (Series 2100 only) |

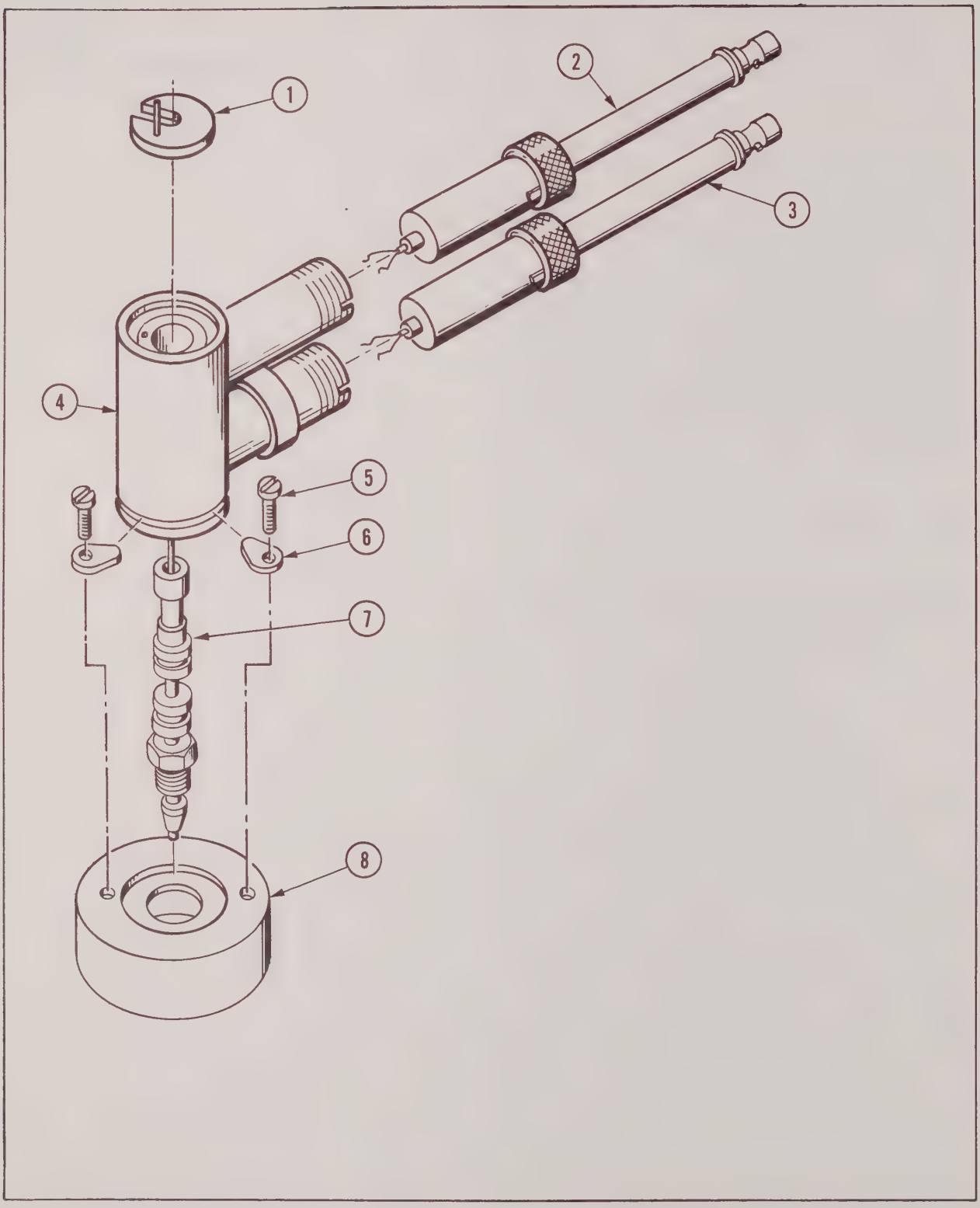
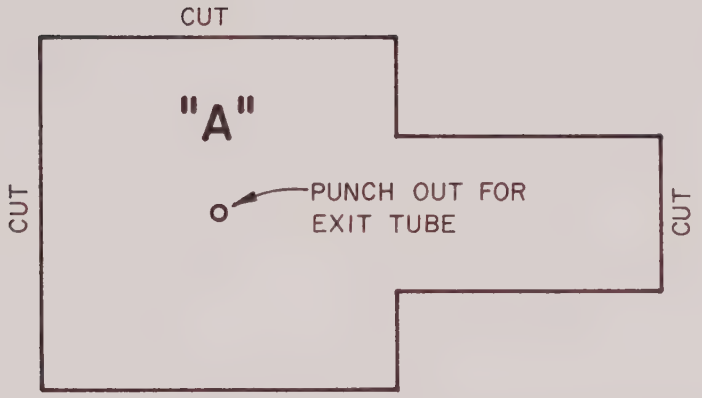
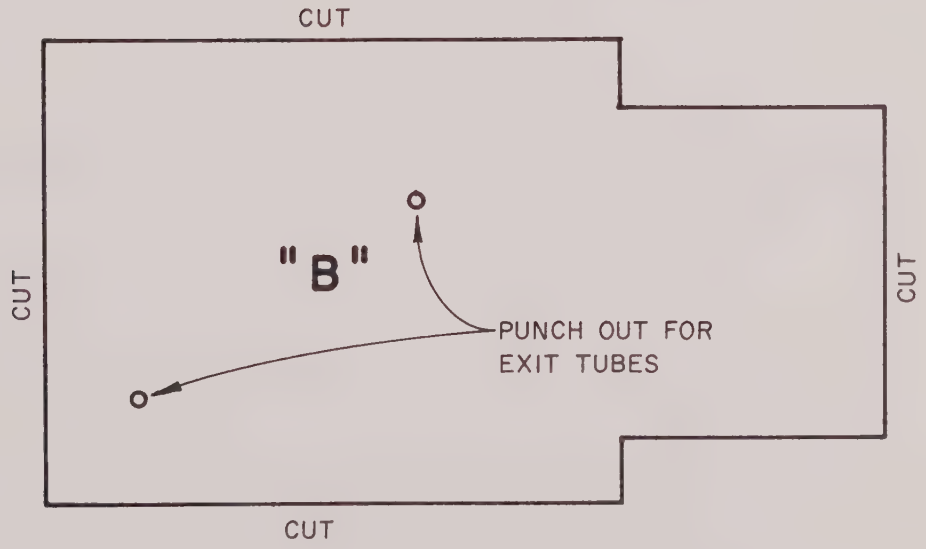



FIGURE 10-1 Sc$^3$H ECD ILLUSTRATED PARTS LIST




1400

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specialists in chromatography and data handling systems

# $^{63}Ni$
# ELECTRON CAPTURE DETECTOR

# INSTRUCTION MANUAL

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Manufactured in the United States of America

PUBLICATION NO. 85-001118-14

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## SAFETY PRECAUTIONS AND OPERATING INSTRUCTIONS

This instruction manual is provided to help you establish operating conditions which will permit efficient use of your equipment. You will notice that the manual describes special considerations and precautions in the form of notes, cautions, and warnings, that not only aid you in obtaining the best performance from your equipment but in calling to your attention potential hazards (e.g., electrical, mechanical, etc.) that might cause personal injury, and how to avoid these hazards. It is important, therefore, that you operate your equipment in accordance with this instruction manual and additional information which may be provided by Varian Instrument Division, Aerograph® Products. Address any questions regarding the safe and proper use of your instrument to:

Varian Instrument Division, Aerograph® Products
611 Hansen Way
Palo Alto, California 94303

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## SPARE PARTS AVAILABILITY

It is the policy of the Varian Instrument Division to provide operational spare parts for any instrument and major accessory for a period of five (5) years after shipment of the final production run of that instrument. Spare parts will be available after this five (5) year period but on an "as available" basis.

Operational spare parts are defined as those individual electrical or mechanical parts that are susceptible to failure during their normal operation. Examples include resistors, inductors, capacitors, semi-conductor devices of all kinds, relays, lamps, temperature probes, detector elements, motors, etc.

Sheet metal parts, structural members or assemblies, and castings, printed circuit cards, and functional modules are normally capable of being rebuilt to like-new condition throughout their useful life and therefore will be supplied on an "as available" basis only after the final production run of the instrument.

The repair and/or reconditioning of instruments and accessories is handled on a time and materials basis after expiration of the Warranty period. Please contact your nearest Varian Instrument Division Sales and Service Office for a quotation.

6/20/1972

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# SECTION 1
# INTRODUCTION

## 1.1 GENERAL

The Varian Aerograph™ $^{63}Ni$ Electron Capture Detector ($^{63}Ni$ ECD) is selectively sensitive to electron-absorbing compounds. Using a Beta source of $^{63}Ni$, electron-absorbing compounds, such as chlorinated hydrocarbons, can be detected at the picogram level with a selectivity of 500,000 to 1 over normal hydrocarbons. Although a theoretical understanding of the electron capture phenomenon is not completely understood, the detector will meet most application requirements by following the installation and operating instructions outlined in this manual.

NOTE

**The Atomic Energy Commission (AEC) and Agreement State Agencies require that the $^{63}Ni$ ECD be tested for leakage every six months. If you are not equipped to perform a leak test, contact one of the following companies or other authorized licensee.**

**Trapelo/West, 2030 Wright Ave., Richmond, Calif. 94804; Radiation Detection Co., 385 Logue Ave., Mountain View, Calif. 94042; Ohmart Corp., 4241 Allendorf Drive, Cincinnati, Ohio 45209.**

## 1.2 SPECIFICATIONS

**Sensitivity**
DC Mode: $5 \times 10^{-14}$ g/sec ($CCl_4$).
Pulse Mode: Approximately the same as the DC Mode.

**Linearity**
$\geqslant 50$

**Standing Current**
$\geqslant 1 \times 10^{-9}$ A

**Noise Level**
$\approx 2 \times 10^{-12}$ A peak-to-peak.

**Ionization Source**
$^{63}Ni$ Beta Source (8.5 mCi).

**Upper Limit Temperature**
400°C

**Polarizing Voltage**
-90 V

**Specificity**
500,000 to 1, or greater.

**Carrier Gas**
High-purity Nitrogen (oxygen and water vapor free) or Argon + 10% $CH_4$.

# SECTION 2
# INSTALLATION

## 2.1 GENERAL

The $^{63}$Ni Electron Capture Detector ($^{63}$Ni ECD) can be installed in any of the following instruments: 1200, 1400, 1700, 1800, 2100, 2700, and 2800 Series. The $^{63}$Ni ECD is purchased as a kit and includes all necessary installation hardware. Table 2-1 identifies the proper Kit Number for each instrument series.

TABLE 2-1
KIT NUMBER/INSTRUMENT IDENTIFICATION

| Kit Number | Instrument Series |
|---|---|
| 02-000770-00 | 1200 |
| 02-000965-00 | 1400 |
| 02-000965-00 | 1700 |
| 02-000965-00 | 1800 |
| 02-000770-00 | 2100 |
| 02-000965-00 | 2700 |
| 02-000965-00 | 2800 |

## 2.2 UNPACKING

When unpacking the $^{63}$Ni ECD Kit, carefully check to ensure that all components ordered have been supplied. Refer to the accessory packing list and the installation instructions in this manual to identify the parts. If there are any discrepancies, notify Varian Aerograph.

NOTE

**If any damage is found, immediately notify the Carrier and Varian Aerograph. Do not return the Kit to the factory without written authorization from Varian Aerograph. Include a copy of the authorization with the Kit when returning Kit to the factory.**

## 2.3 LOCATION

The instrument in which the $^{63}$Ni ECD is installed should be located where it will not be subjected to abrupt temperature changes, excessive vibration, or strong air currents (such as under a heater or air conditioning vent). Ensure adequate space is available for necessary gas supplies.

## 2.4 GAS CONNECTIONS

Refer to the following paragraphs for the gas supply and controls required to operate the $^{63}$Ni ECD.

NOTE

**Contact your Varian Aerograph Sales Representative for flow measuring and flow control accessories required to install your gas supply.**

### 2.4.1 Carrier Gas Supply

The required carrier gas supply plumbing is illustrated in Figure 2-1. All of the components shown in Figure 2-1 are necessary except for the flow diverter. However, the flow diverter does allow the carrier gas to be monitored without disconnecting instrument plumbing. The components comprising the carrier gas supply are described in the following paragraphs.

- **Gas Supply**

The gas supply is a standard compressed gas cylinder. Nitrogen or argon + 10% $CH_4$ can be used for carrier gas although nitrogen is preferred.

- **Two-Stage Pressure Regulator**

The two-stage pressure regulator monitors the gas cylinder pressure and regulates the second stage pressure. Set the second stage pressure for 60 to 100 psi.

- **Shut-Off Valve**

The shut-off valve is used to turn the carrier gas on and off as required. The shut-off valve eliminates the need for using the flow controller valve as an

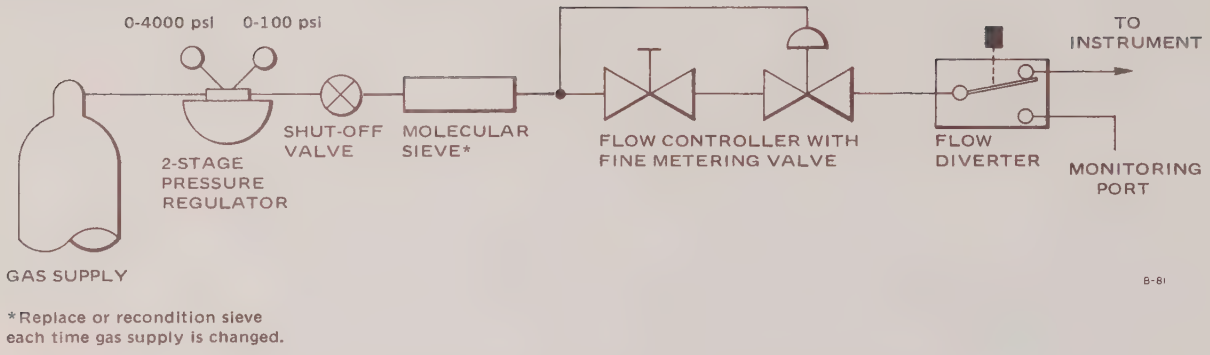



FIGURE 2-1 $^{63}$Ni CARRIER GAS SUPPLY PLUMBING DIAGRAM

on-and-off valve which can damage the controller valve seat.

• Flow Controller

The flow controller maintains a constant gas flow through the column as the column head pressure varies. The desired carrier gas flow rate is set with the fine metering valve and then maintained by the flow controller. Ensure the flow controller pressure differential, working pressure, flow range, and flow accuracy specifications meet your needs.

— CAUTION —

**Do not use the flow controller valve as an on-and-off valve. Tightening the valve fully clockwise may damage the valve seat.**

• Flow Diverter

The flow diverter is a manually operated three-way valve. In the position shown in Figure 2-1, the carrier gas is connected to the instrument; whereas, in the opposite position, the gas is vented to atmosphere via the monitoring port. The flow rate can be checked by connecting a soapbubble flowmeter to the monitoring port.

## 2.5 INSTALLATION PROCEDURE

This section of the manual comprises the installation procedure for the $^{63}$Ni ECD.

The 1700, 1800, 2700, and 2800 Series Instruments have two ionization detector bases. The $^{63}$Ni ECD can be installed on either base; however, the $^{63}$Ni ECD output signal polarity is the reverse of a flame ionization detector. Therefore, the "B" channel position is preferred since the proper signal polarity is available on the "B" channel recorder cable when the Electrometer MODE switch is set in the proper position. If the detector is installed in the "A" channel position, reverse the recorder cable leads at the rear of the instrument or the recorder.

The 2100 Series Instruments can have two or four detector bases; one "A" channel and one "B" channel or two "A" channels and two "B" channels. The signal polarity parameters are the same as the 1700, 1800, 2700, and 2800 Series Instruments.

The 1400 Series Instruments have only one detector base. When the electrometer CELL VOLTAGE switch is set to the EC position, the recorder cable leads are automatically reversed. Therefore, the recorder cable leads do not require any change.

Since there are two $^{63}$Ni ECD Kits, there are two installation procedures. Using the appropriate installation procedure, install the $^{63}$Ni ECD as follows:

### 2.5.1 1200 and 2100 Series Instruments

1) Set appropriate electrometer CELL VOLTAGE switch to the OFF position.

**WARNING**

**To prevent a possible shock hazard, ensure that the cell voltage is turned off and that the cell voltage cable is disconnected from the detector tower.**

2) Turn off power to the detector oven. Allow the detector base and detector tower to cool before handling.

3) To gain access to the existing detector on Series 1200 Instruments, open the column-oven door; on Series 2100 Instruments, unfasten the two thumbscrews on the detector-oven lid, then raise the lid.

4) Remove the existing detector tower and tip assembly.

NOTE

**When installing the $^{63}$Ni ECD in a 1200 Series Instrument, mount the temperature-limit-switch actuator arm to the tower as shown in Figure 2-2. The actuator arm actuates a microswitch which allows the detector oven to heat above 225°C. The 2100 Series Instruments do not require the actuator arm.**

5) While holding the $^{63}$Ni cell assembly by the mounting nut, clean the glass sections of the assembly with tissue and hexane.

— CAUTION —

**Do not touch glass sections of cell assembly. Fingerprints provide high-resistance current paths which can cause noise, high "observed" standing current, and low sensitivity.**

6) Install the $^{63}$Ni cell assembly on the detector base as shown in Figure 2-2.

7) Secure the $^{63}$Ni cell assembly to the detector base by tightening the mounting nut clockwise.

— CAUTION —

**When tightening the mounting nut, be careful the wrench does not slip and break the cell assembly.**

8) Remove the tower cap from the detector.

9) Loosen the knurled nuts on the anode and cathode electrodes, then retract both electrodes about one inch.

10) Set the detector tower onto the detector base. Ensure the tower is properly seated on the raised portion of the detector base. If the $^{63}$Ni ECD is being installed in a 1200 Series Instrument, ensure that the actuator arm is actuating the temperature limit switch.

11) Insert the mounting screws through the tower brackets and into the detector base mounting tabs as shown in Figure 2-2.

12) Tighten the mounting screws until the detector is firmly held in place.

13) Reinstall the anode and cathode electrodes in the detector tower as shown in Figure 2-3 (note that the two-pin connector electrode goes in the upper position). Ensure alignment tabs on the electrode housings (part of tower) are engaged with the slots in the electrode body before tightening the knurled nuts.

— CAUTION —

**Glass sections of the cell assembly are fragile. Use caution when slipping electrode clips onto the metal sections of the cell assembly.**

14) Place the cap on the tower.

15) Place the insulated tower cover on the detector. If required, adjust the insulation inside the tower cover, and the position of the cover so that the cover slips down over the tower.

NOTE

***AEC regulations require* that the radioactive emblem on the insulated tower cover be visible while installed.**

16) Reinstall covers, insulation, lids, and other items removed to gain access to the detector base.

17) Connect the anode (signal) and cathode (cell voltage) cables to the appropriate electrodes.

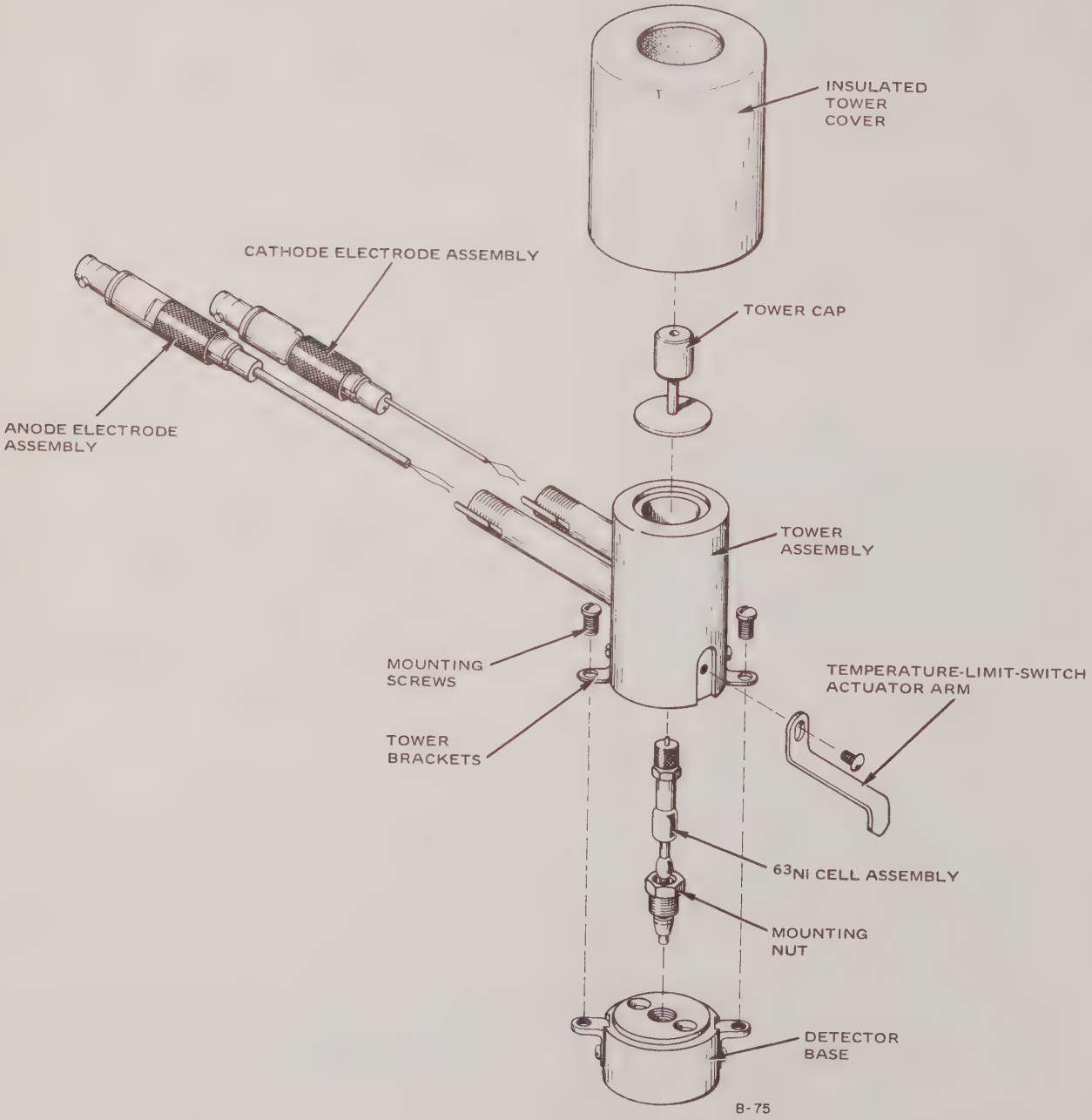
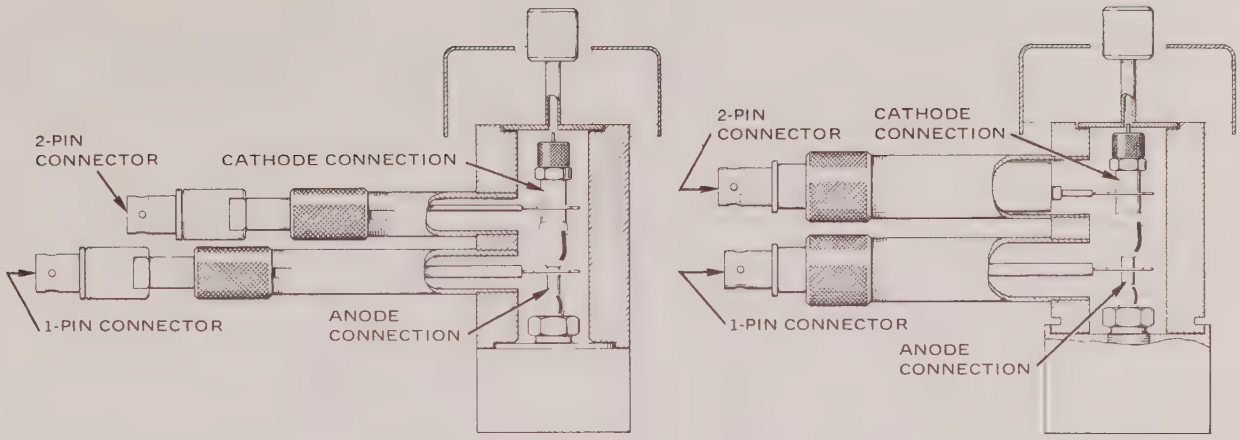



FIGURE 2-2 $^{63}$Ni ECD INSTALLATION ON 1200 AND 2100 SERIES INSTRUMENTS




1200 and 2100 Series Instruments

1400, 1700, 1800, 2700, and 2800 Series Instruments

FIGURE 2-3 ANODE AND CATHODE ELECTRODE POSITIONING

Note that the cell voltage cable has a 2-pin connector and connects to the upper electrode; whereas, the signal cable has a 1-pin connector and connects to the lower electrode.

18) Since the $^{63}Ni$ ECD operation does not require $H_2$, disconnect the $H_2$ source from the instrument.

NOTE

**The 2100 Series Instruments have only one $H_2$ inlet for all the detector positions. Therefore, it may be necessary (when operating an ECD and FID simultaneously) to use the individual HYDROGEN needle valves to isolate the $^{63}Ni$ ECD position from the $H_2$ supply.**

19) Purge the $H_2$ line between the detector and $H_2$ inlet fitting with $N_2$. Disconnect the $N_2$ and cap off the $H_2$ inlet fitting. Capping the $H_2$ inlet fitting prevents carrier gas and sample from leaking out the $H_2$ line. Also, air is prevented from back diffusing into the $^{63}Ni$ cell assembly via the $H_2$ line (since oxygen has an affinity for free electrons, air will cause a decrease in the background current and sensitivity).

NOTE

**Temperature changes caused by room drafts and air conditioning ducts may result in random baseline fluctuations and detector instability. To reduce temperature change effects, a piece of insulation can be placed over the exposed portion of the detector.**

20) The $^{63}Ni$ ECD installation is completed. Refer to Section 4 of this manual for the operating procedure.

**2.5.2 1400, 1700, 1800, 2700, and 2800 Series Instruments**

1) Set appropriate electrometer CELL VOLTAGE switch to the OFF position.

WARNING

**To prevent a possible shock hazard, ensure that the cell voltage is turned off and that the cell voltage cable is disconnected from the detector tower.**

2) Turn off power to the detector oven. Allow the detector base and detector tower to cool before proceeding with the installation.

3) To gain access to the existing detector on Series 1400 Instruments, open the column-oven door; on Series 1700, 1800, 2700, and 2800 Instruments, remove the snap-on detector-oven lid, insulation pad, and oven-cover plate.

4) Remove the existing detector tower and tip assembly.

5) While holding the $^{63}Ni$ cell assembly by the mounting nut, clean the glass sections of the assembly with tissue and hexane.

— CAUTION —

**Do not touch glass sections of cell assembly. Fingerprints provide high-resistance current paths which can cause noise, high "observed" standing current, and low sensitivity.**

6) Install the $^{63}Ni$ cell assembly on the detector base as shown in Figure 2-4.

7) Secure the $^{63}Ni$ cell assembly to the detector base by tightening the mounting nut clockwise.

— CAUTION —

**When tightening the mounting nut, be careful the wrench does not slip and break the cell assembly.**

8) Remove the tower cap from the detector.

9) Loosen the knurled nuts on the anode and cathode electrodes, then retract both electrodes about one inch.

10) Set the detector tower onto the detector base. Position the base of the tower inside the detector base recess.

11) Rotate the retaining tabs clockwise until they engage with the slot in the bottom of the detector tower as shown in Figure 2-4.

12) Tighten the mounting screws until the detector is firmly held in place.

13) Reinstall the anode and cathode electrodes in the detector tower as shown in Figure 2-3 (note that the 2-pin connector electrode goes in the upper position). Ensure alignment tabs on the electrode housings (part of tower) are engaged with the slots in the electrode body before tightening the knurled nuts.

— CAUTION —

**Glass sections of the cell assembly are fragile. Use caution when slipping electrode clips onto the metal sections of the cell assembly.**

14) Place the cap on the tower.

15) Place the insulated tower cover on the detector.

NOTE

***AEC regulations require* that the radioactive emblem on the insulated tower cover be visible while installed.**

16) Reinstall covers, insulation, lids, and other items removed to gain access to the detector base.

17) Connect the anode (signal) and cathode (cell voltage) cables to the appropriate electrodes. Note that the cell voltage cable has a 2-pin connector and connects to the upper electrode; whereas, the signal cable has a 1-pin connector and connects to the lower electrode.

18) Since the $^{63}$ Ni ECD operation does not require $H_2$, disconnect the $H_2$ source from the instrument.

NOTE

**The 1700, 1800, 2700, and 2800 Series Instruments have only one $H_2$ inlet for all the detector positions. Therefore, it may be necessary (when operating an ECD and FID simultaneously) to use the individual HYDROGEN needle valves to isolate the $^{63}Ni$ ECD position from the $H_2$ supply.**

19) Purge the $H_2$ line between the detector and $H_2$ inlet fitting with $N_2$. Disconnect the $N_2$ and cap off the $H_2$ fitting. Capping the $H_2$

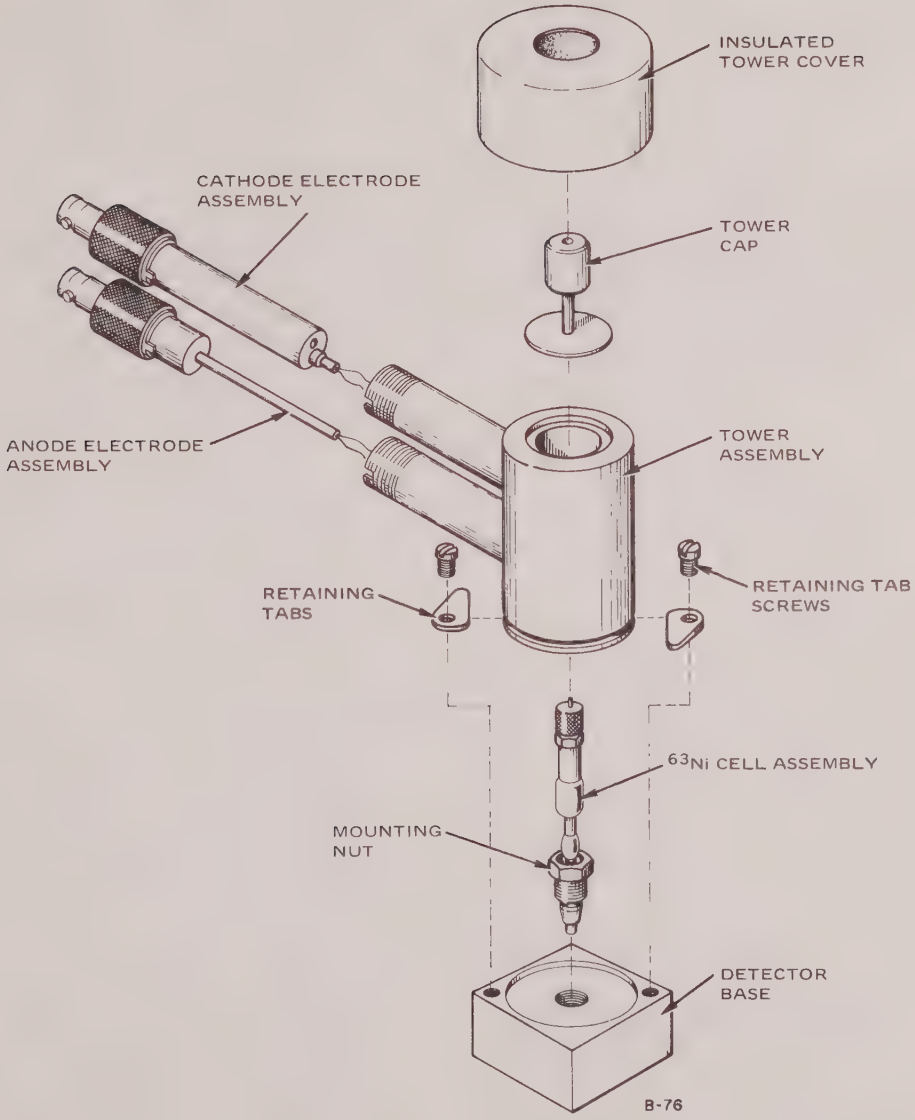



FIGURE 2-4 $^{63}$Ni INSTALLATION ON 1400, 1700, 1800, 2700 AND 2800 SERIES INSTRUMENTS

inlet fitting prevents carrier gas and sample from leaking out the $H_2$ line. Also, air is prevented from back diffusing into the $^{63}$Ni cell assembly via the $H_2$ line (since oxygen has an affinity for free electrons, air will cause a decrease in the background current and sensitivity).

NOTE

**Temperature changes caused by room drafts and air conditioning ducts may result in random baseline fluctuations and detector instability. To reduce temperature change effects, a piece of insulation can be placed over the exposed portion of the detector.**

20) The $^{63}$Ni ECD installation is completed. Refer to Section 4 of this manual for the operating procedure.

## 2.6 REMOVAL PROCEDURE

The $^{63}$Ni ECD must be removed and stored in accordance with *AEC regulations.* Therefore, it is necessary to comply with the following instructions when removing the $^{63}$Ni ECD.

1) Set the appropriate electrometer CELL VOLTAGE switch to the OFF position.

2) Disconnect the cathode and anode cables at the detector.

3) Loosen the knurled nuts on the anode and cathode electrodes. Remove both electrodes as shown in Figure 2-2 or 2-4.

4) Remove necessary lids, covers, and insulation to gain access to the detector base.

5) Locate the detector retaining tabs, or screws, at the base of the detector tower.

6) Loosen or remove, whichever is appropriate, the detector tower retaining screws.

7) Remove the detector tower by lifting straight up. Avoid hitting the cell assembly when removing the tower, otherwise, glass sections may break or crack.

8) Carefully loosen the mounting nut to free the cell assembly from the detector base.

— CAUTION —

**When loosening the mounting nut, be careful the wrench does not slip and break the cell assembly.**

9) Use the mounting nut (ensure nut is not hot) to lift the cell assembly off the detector base.

10) Place the cell assembly in a container with adaquate packing material to prevent breakage.

11) Reassemble the tower and electrodes (tower functions as a protective cover for the electrodes) as shown in Figure 2-3.

12) Package the cell assembly and associated parts together in a single package. *AEC regulations require* a radioactive emblem to be placed on the outside of the package.

WARNING

***Never try to gain access to the $^{63}$Ni source in the cell assembly.*** **The cell assembly is registered with the *AEC* as a sealed-source; therefore, removing the knurled cap on the cell assembly is in violation of *AEC regulations.***

# SECTION 3
# PRINCIPLES OF OPERATION

## 3.1 GENERAL

The $^{63}$Ni Electron Capture Detector ($^{63}$Ni ECD) can be operated with a constant or pulsing polarizing voltage. However, there is little, if any, benifit derived from pulsing the Varian Aerograph $^{63}$Ni ECD. Also, the expense for pulsing equipment far exceeds any benefits that may be obtained from the pulsing method; therefore, only the constant (DC) polarizing voltage method will be discussed in this manual.

## 3.2 BASIC PRINCIPLES OF OPERATION

Refer to Figure 3-1 for the following discussion of the $^{63}$Ni ECD operation.

The radioactive foil ($^{63}$Ni) emits $\beta$ particles with a maximum energy of $\approx$ 67 KeV. As the $\beta$ particles travel through the carrier gas, they ionize the carrier gas (nitrogen) and produce positive ions and secondary electrons. The negative cell voltage applied to the cathode connection causes the secondary electrons (free electrons) to be driven towards the anode connection. The free electrons are collected at the anode and produce a steady background (standing) current. When a sample compound with an affinity for free electrons enters the detector, the standing current is reduced. The amount of reduction is a function of the sample concentration and the electron affinity ("capture coefficient") of the compound. The decrease in standing current is sensed by an electrometer through the anode connection. This signal is then amplified to a sufficient level to operate a potentiometer strip-chart recorder.

## 3.3 OPERATIONAL PARAMETERS

A complete understanding of the $^{63}$Ni ECD operational parameters is necessary to properly operate the detector. The following discussion will acquaint the operator with the $^{63}$Ni ECD operational characteristics.

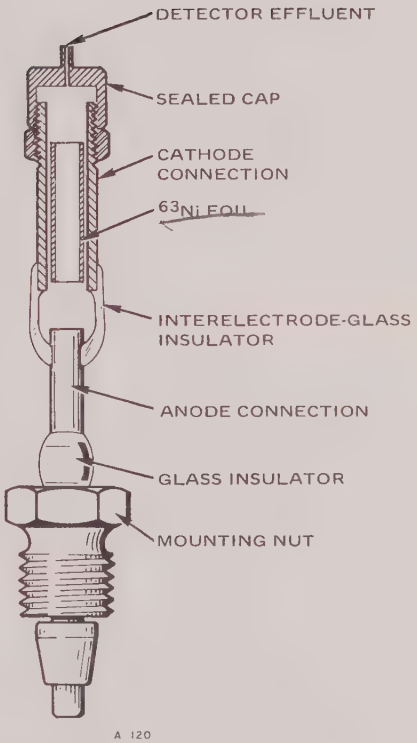



FIGURE 3-1 CROSS-SECTIONAL VIEW OF $^{63}$Ni CELL ASSEMBLY

### 3.3.1 General

The $^{63}$Ni ECD operation is dependent on the ionization of the nitrogen carrier gas by the radioactive foil $\beta$ particles. This ionization (standing) current is an indication of the detector sensitivity, and is the most important parameter of the detector operation. Therefore, the operator must be aware of which parameters affect the standing current. The following paragraphs describe these parameters and how to control them for optimum detector operation.

### 3.3.2 Standing Current

Standing current is a measurement of the rate of collection of free electrons formed by the ionization of the nitrogen carrier gas. This current is defined as the "true" standing current. As shown in Figure 3-2, the "true" standing current decreases as the foil temperature increases. This decrease in current is due to the carrier gas density decreasing as the detector temperature increases, not because the foil temperature increased. For the convenience of the operator, the foil temperature rather than the gas density is used to illustrate temperature affects on the standing current. However, at the same time, the interelectrode-glass insulator resistance is decreasing and provides a second current path. The result is a "false" standing current as shown in Figure 3-2. Therefore, the standing current seen by the operator is the sum of the "true" and "false" standing currents. This sum is shown as the "observed" standing current in Figure 3-2.

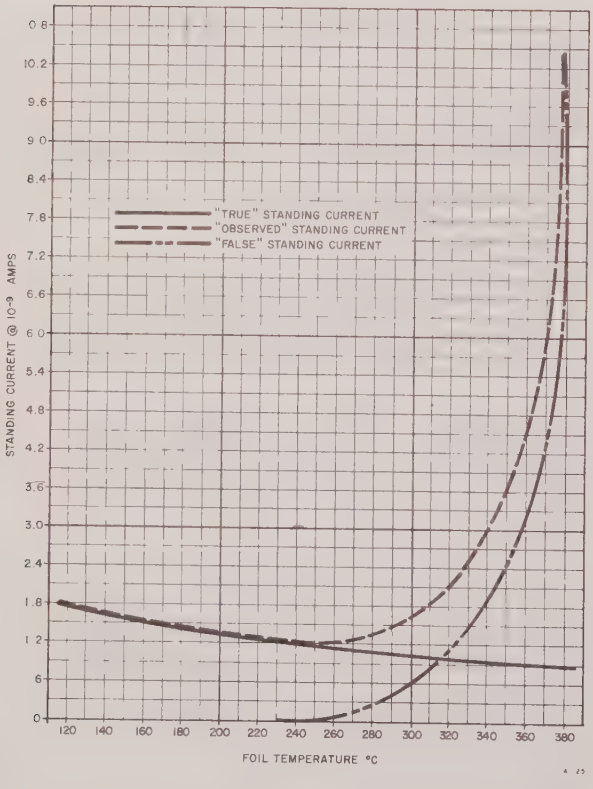



FIGURE 3-2 $^{63}$Ni ECD "TRUE" STANDING CURRENT

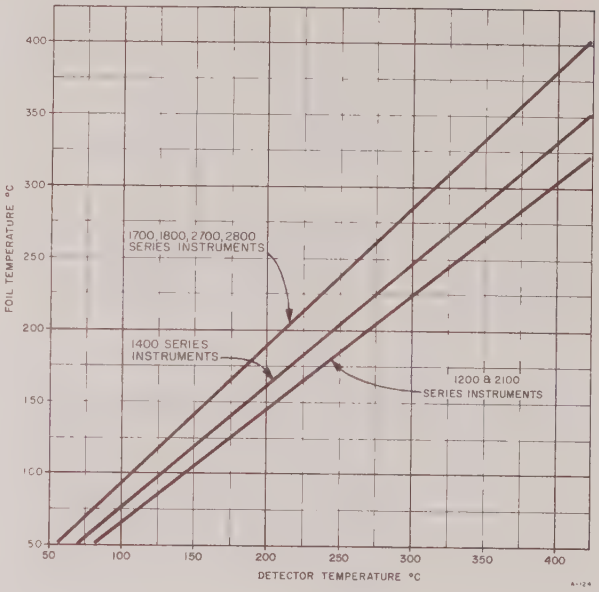

1700, 1800, 2700, 2800 SERIES INSTRUMENTS
1400 SERIES INSTRUMENTS
1200 & 2100 SERIES INSTRUMENTS
FOIL TEMPERATURE °C
DETECTOR TEMPERATURE °C

FIGURE 3-3 FOIL TEMPERATURE Vs DETECTOR TEMPERATURE

More specific data on standing current is described in an article by Martin Scolnick, "The Concentric Cylinder Electron Capture Detector Standing Current Studies in the DC and Pulsed Voltage Modes", in the *Journal of Chromatographic Science,* 7,263 (1969).

### 3.3.3 Foil Temperature Vs Detector Temperature

The radioactive foil temperature with respect to the detector temperature is illustrated in Figure 3-3. The detector temperature is read out on the pyrometer located on the Instrument Main Control Panel. Since there are two distinctly different detector ovens, a typical curve is shown (by instrument Series Number) for the two types of ovens. As shown in Figure 3-4, the foil temperature has a direct affect on the standing current. Therefore, when selecting the detector-oven temperature, keep in mind the foil temperature is less than the detector temperature.

### 3.3.4 Standing Current Vs Foil Temperature

Figure 3-4 illustrates the change in "observed" standing current as the foil temperature is increased. The illustration indicates that the foil temperature (see Figure 3-3 for corresponding detector

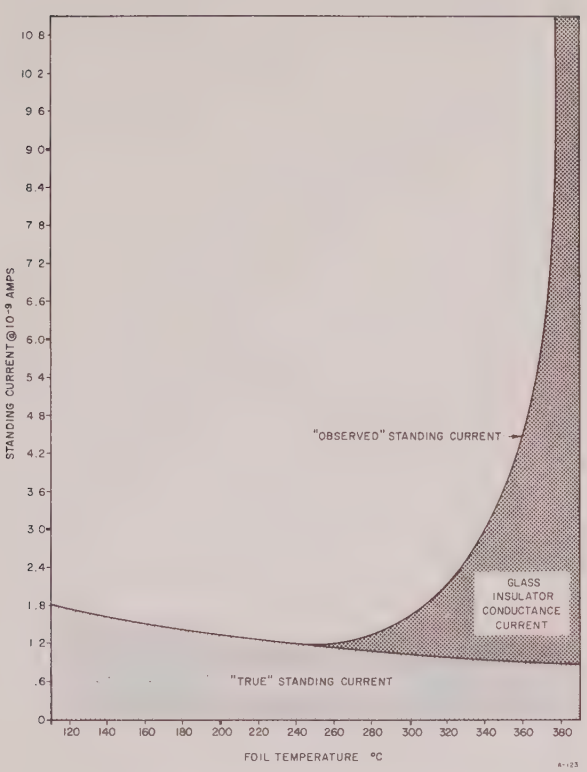



FIGURE 3-4 STANDING CURRENT Vs FOIL TEMPERATURE

temperature) has minimal affect on the "observed" standing current below 250°C; whereas, above 250°C, the interelectrode-glass insulator resistance decreases sufficiently to radically change the "observed" standing current. However, only the "true" standing current is usable. Current in excess of this value, due to conductance across the glass insulator, does not improve the detector sensitivity or linearity, but does increase noise and detector instability.

### 3.3.5 Carrier Gas Flow Rate

If the chromatography system is leak tight, the carrier gas flow rate has little affect on the standing current of the $^{63}Ni$ ECD. The carrier gas flow rate should be determined from chromatographic considerations such as peak separation and column technology. The recommended carrier gas flow rates for typical analyses is 15 to 40 ml/min for 1/8-in columns, and 40 to 60 ml/min for 1/4-in columns. Flow rates as low as 10 ml/min have been used without back diffusion of oxygen into the cell assembly through the exit tube, but are not generally recommended.

**— CAUTION —**

**To prevent detector contamination, always use high-purity nitrogen (oxygen and water vapor free) for the carrier gas.**

### 3.3.6 Detector Temperature

The general rule is to operate the detector temperature 50°C higher than the column-oven temperature. This minimizes condensation of column and sample contaminants on the radioactive foil as they elute from the column. Condensates on the radioactive foil cause the detector standing current and sensitivity to decrease.

**— CAUTION —**

***Always* bring the detector up to operating temperature before setting the carrier gas flow rate, or heating the column oven and injector. Otherwise, the carrier gas will carry contaminants into the detector where they will condense on the radioactive foil.**

### 3.3.7 Linear Range

The linear range (detector linearity) of a detector is the ratio of the largest to the smallest concentration of sample within which the detector is linear. The smallest concentration is that amount which results in a response twice the noise level. The largest amount would then be the highest concentration that results in a response directly proportional (± 2%) to the smallest concentration.

It should be kept in mind that the detector responds to the amount of sample per time unit (i.e., g/sec). Therefore, a compound with a short retention time can be detected at lower concentrations than compounds with longer retention times. Also, this means the largest amount that can be detected, within the linear range, is partially dependent on the retention time and column-oven temperature. Therefore, to achieve optimum quantitative results, the linear dynamic range of a new sample should be determined at specific temperatures and flow rates.

# SECTION 4
# OPERATION

## 4.1 GENERAL

The purpose of this section of the manual is to familiarize the operator with the function and operation of the $^{63}Ni$ Electron Capture Detector ($^{63}Ni$ ECD). Before operating the detector, read Sections 3 and 4 to become familiar with the $^{63}Ni$ ECD terminology and characteristics.

## 4.2 DETECTOR CHARACTERISTICS

The following information will familiarize the operator with the $^{63}Ni$ ECD characteristics. Understanding the detector characteristics will assist the operator in selecting the proper operational parameters.

### 4.2.1 Linearity

As shown in Figure 4-1, the $^{63}Ni$ ECD response to Aldrin and Lindane is linear over an $\approx$ 50 fold range. Also, Table 4-1 lists the typical minimum and maximum detectable quantities for several other pesticides.

TABLE 4-1

| Compound | Minimum | Maximum |
|---|---|---|
| Dieldrin | 1.9 pg | 95 pg |
| Heptachlor | 2.1 pg | 105 pg |
| Methyl Parathion | 27.6 pg | 1,400 pg |
| Di-Syston | 64.0 pg | 3,200 pg |

### 4.2.2 Sensitivity

One of the characteristics of the $^{63}Ni$ ECD is the relationship between the sample sensitivity and the cathode potential (cell voltage). As shown in Figure 4-2, the detector response, for injections of Lindane, reaches a plateau once the cell voltage exceeds 60 Vdc. Since the $^{63}Ni$ ECD is operated with a 90 Vdc cell voltage, the detector operates well within the plateau region.

### 4.2.3 Specificity

The selectivity of response of the $^{63}Ni$ ECD is variable, depending on the detected compounds' affinity for free electrons ("capture coefficient"). Generally the detector has a discrimination factor

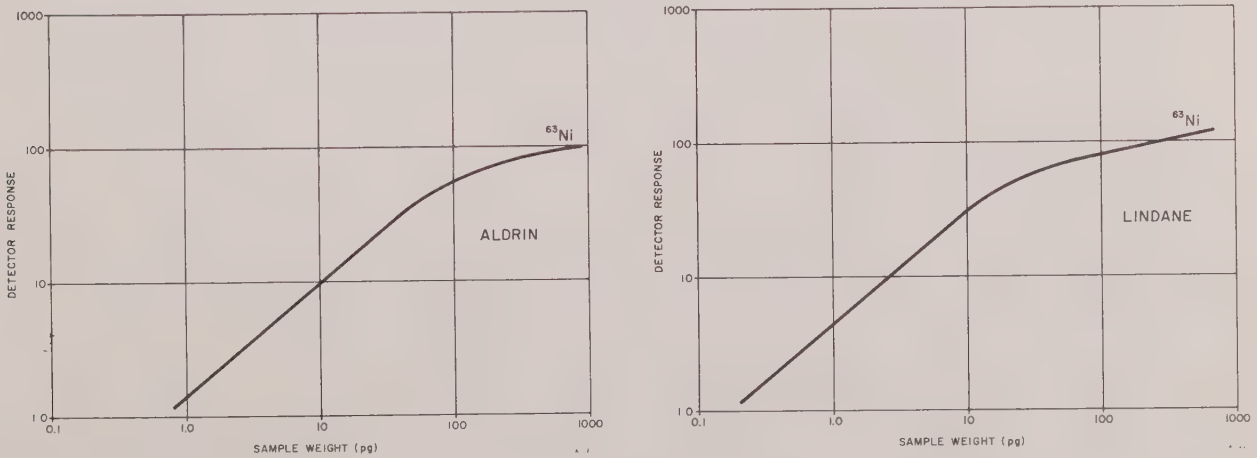



FIGURE 4-1 $^{63}Ni$ ECD LINEARITY

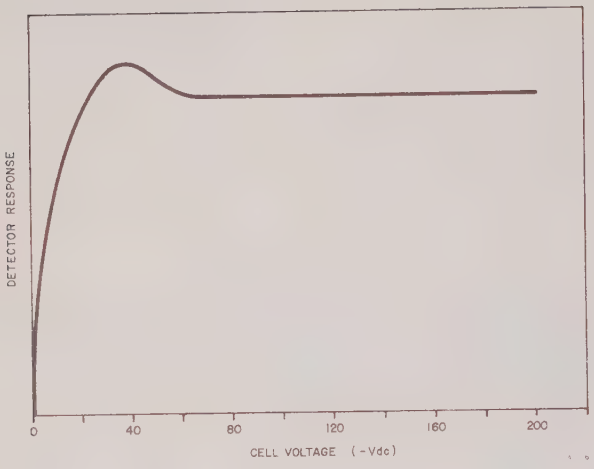



FIGURE 4-2 $^{63}$Ni ECD SENSITIVITY Vs CELL VOLTAGE

of 500,000 to 1 against normal hydrocarbons. An example of the $^{63}$Ni ECD selectivity against normal hydrocarbons is illustrated in Figure 4-3. A sample of peppermint oil (natural) was simultaneously analyzed by a $^{63}$Ni ECD and FID to check for components not detectable by just one of the detectors.

### 4.2.4 $^{63}$Ni Isotope Characteristics

The $^{63}$Ni isotope characteristics are listed in Table 4-2.

TABLE 4-2
$^{63}$Ni ISOTOPE CHARACTERISTICS

| | |
|---|---|
| Half Life | 125 years |
| Type of Decay | -$\beta$ |
| Particle Energy | 67 KeV |
| Specific Activity | 5 Ci/g |
| Physical Properties | solid |
| Foil Temperature Limitations | 400°C |
| Radio Toxicity | moderate |

### 4.2.5 "True" Standing Current

The $^{63}$Ni ECD "true" standing current is typically $\geq 1 \times 10^{-9}$ A at temperatures $\leq 250^{\circ}$C. As discussed in paragraph 3.3.2, there are "true", "false", and "observed" standing currents. The "true" and "observed" standing currents are generally the same below 250°C; however, above 250°C the "observed" current increases exponentially while the "true" standing current decreases. Since the "observed" current is not always representative of the "true" current, other parameters, visible to the operator, must be used to determine whether the detector is operating properly (i.e., temperatures, sensitivity, and the "observed" standing current relationship to these parameters). Section 5 of this manual covers typical problems encountered when the standing current and detector operation do not maintain the expected relationship.

### 4.2.6 Noise Level

The $^{63}$Ni ECD noise is typically $2 \times 10^{-12}$ A peak-to-peak. The noise generally increases as the

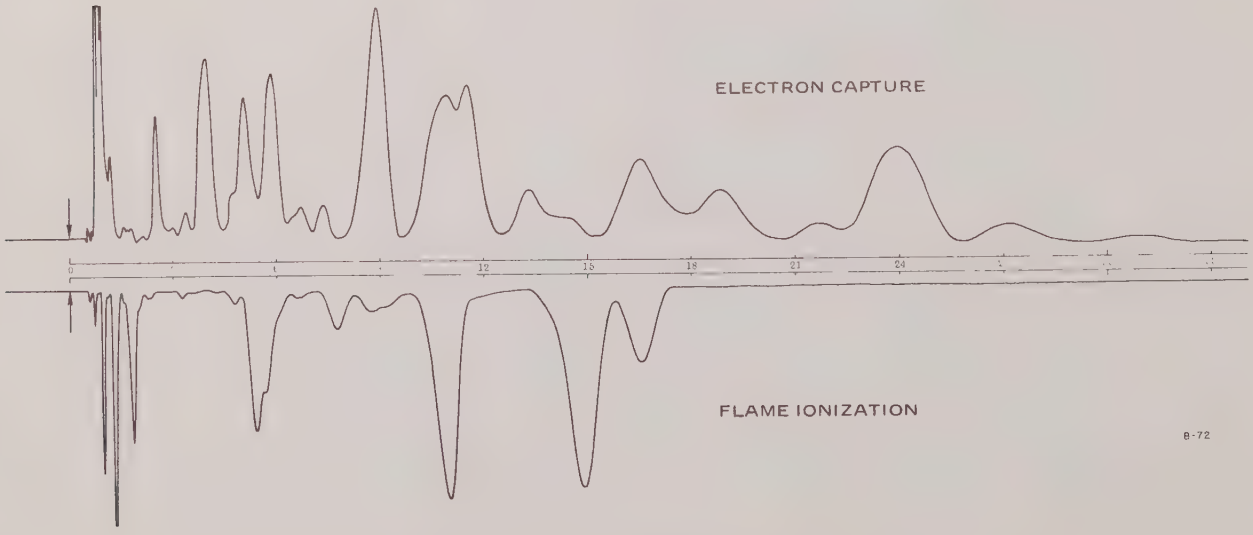



FIGURE 4-3 $^{63}$Ni ECD — FID RESPONSE COMPARISON

foil temperature increases. However, an abnormal level of noise is generally associated with a contaminated chromatographic system.

#### 4.2.7 Stability

The $^{63}$Ni ECD stability is very temperature dependent, especially above 250°C (foil temperature). Therefore, the detector must be protected from external temperature gradients (e.g., air conditioning ducts, abrupt room temperature changes, etc.). Also, the Series 1200 Instruments use a bi-metalic thermostat to control the detector-base heater which can result in baseline cycling. That is, as the thermostat cycles, the detector temperature cycles, and the foil temperature cycles. The resulting changes in the standing current may be sufficient to cycle the baseline. To minimize the baseline cycling to an acceptable level, replace the existing detector-base heater (150 W) with a 100 W cartridge heater.

### 4.3 PREOPERATIONAL SETUP

The preoperational setup is required to prepare your instrument for use with a $^{63}$Ni ECD. The detector requires a contaminant free chromatographic system; otherwise, the detector radioactive foil will be contaminated and the standing current will be reduced to an unacceptable level.

Decontaminate your instrument as follows:

1) Ensure a detector is not installed on the detector base to be used.

2) Cap off the injector. Use an injector blank-off nut or aluminum foil and a septum.

3) Install the column that has been selected for the analyses.

— CAUTION —

**Do not connect the column effluent to the detector or crossover. Venting column effluent to atmosphere prevents contaminants from entering the detector.**

4) Set the carrier gas for some nominal flow rate. The carrier gas will purge the column during the conditioning period.

5) Determine the maximum operating temperature for the column packing liquid phase being used. Table 4-3 lists some of the more commonly used liquid phases and their maximum temperatures. The maximum temperatures for other liquid phases can be found in the Varian Aerograph *Instrument and Accessories Catalog.*

6) Set the DETECTOR SAFETY LIMIT switch to the OFF position (2100 Series Instruments only — switch located on Instrument Main Control Panel).

TABLE 4-3
COLUMN CONDITIONING TEMPERATURES

| Liquid Phase | Maximum Temperature |
|---|---|
| Carbowax 20M | 250°C |
| DC-11 | 300°C |
| DC-200 | 250°C |
| OV-1 | 325°C |
| OV-17 | 325°C |
| OV-101 | 325°C |
| OV-210 | 250°C |
| OV-225 | 250°C |
| QF-1 | 250°C |
| SE-30 | 300°C |
| SE-52 | 300°C |

7) Set the column-oven temperature at the *liquid phase maximum* temperature.

8) Condition the colum for a minimum of 48 hours.

— CAUTION —

**The $^{63}$Ni ECD operation is *very* dependent on the length of time the column has been conditioned. An improperly conditioned column will contaminate the chromatographic system and quench the detector standing current.**

9) Turn off power to the instrument. Allow the column oven, detector base, and injector to cool until their components can be handled.

10) Turn off the carrier gas and remove the analytical column.

11) Install an empty metal column and reestablish the carrier gas flow rate.

— CAUTION —

**Do not use columns with Teflon® ferrules when injector, column, or detector temperatures exceed 250°C. The Teflon ferrules begin to flow at temperatures above 250°C.**

12) Set the column oven, detector base, and injector temperatures at 400°C.

13) Allow the column oven, detector base, and injector to bake out for a minimum of 8 hours.

14) Set the column oven, detector base, and injector temperatures at 300°C.

15) Make 5 injections of 100 µl of distilled water (steam cleans the chromatographic system).

16) Condition several silicone septa at 250°C for 16 hours.

NOTE

**The $^{63}$Ni ECD standing current is quenched by septum bleed. The procedure outlined in Step (16) generally decontaminates the septa adequately; however, it also decreases the number of injections that can be made before the septum leaks.**

17) Turn off power to the instrument. Allow the column oven, detector base, and injector to cool until their components can be handled.

18) Turn off the carrier gas and remove the empty column.

19) Reinstall the conditioned analytical column.

20) Leak check the chromatographic system as follows:

a) Cap off the injector and detector base.
b) Heat chromatographic system to operating temperature.
c) Pressurize the chromatographic system to 65 psi with nitrogen.
d) Turn off the nitrogen supply. The chromatographic system must hold the 65 psi for a minimum of 1 hour.
e) If the system has a leak, take the necessary steps to eliminate the leaks. Refer to the Basic Instrument Manual for more specific details on leak checking the system.
f) Once leaks are eliminated, depressurize the system and remove the injector and detector blank-off nuts.

21) Install a decontaminated septum on the injector.

22) Install the $^{63}$Ni ECD on the decontaminated detector base as described in Section 2 of this manual.

## 4.4 ROUTINE OPERATION

As stated in paragraph 3.1, there is little, if any, advantage to pulsing the cell voltage; therefore, only the DC Mode of operation will be discussed in this paragraph.

Operate the $^{63}$Ni ECD as follows:

1) Set up the instrument as described in paragraph 4.3, PREOPERATIONAL SETUP.

2) Ensure the $^{63}$Ni ECD and carrier gas supply are installed as described in Section 2 of this manual.

3) Set up the desired detector temperature. The detector foil temperature should be ≈ 50°C higher than the desired operating column-oven temperature. Refer to Figure 3-3 for the foil temperature versus detector temperature curves.

— CAUTION —

***Always* bring the detector up to operating temperature before setting the carrier gas flow rate or heating the column oven and injector. Otherwise, the carrier gas will carry contaminants into the detector where they will condense on the radioactive foil.**

4) Set the carrier gas for the desired flow rate.

5) Set up the desired column-oven temperature.

6) Set up the desired injector temperature. The on-column injection method is preferred because the required injector temperature is less than the flash vaporization method (higher the injector temperature, greater the septum bleed).

7) Set the electrometer MODE control to the appropriate position:

   EC: Single Channel Electrometer
   FL-EC: Dual Channel Electrometer

8) Check the $^{63}$Ni ECD standing current as described in paragraph 4.5.

9) When the proper standing current is obtained, set the CELL VOLTAGE control to the EC position.

10) Set the RANGE and ATTENUATOR controls to the desired positions.

11) Use the BUCKING control to set the recorder pen at electrical zero.

12) The $^{63}$Ni ECD is now ready to operate. If the detector is being used continuously, check the standing current everyday.

## 4.5 CHECKING STANDING CURRENT

The standing current must be checked when the detector is first installed and periodically thereafter. The standing current should remain relatively constant. A change in the standing current generally indicates the detector foil temperature is in the interelectrode-glass insulator conductance range, or the foil is contaminated.

The following procedure presumes the $^{63}$Ni ECD is installed, all temperatures are set, and the carrier gas flow rate has been set. Check the standing current as follows:

1) Set the electrometer MODE control to the appropriate position:

   EC: Single Channel Electrometer
   FL-EC: Dual Channel Electrometer

2) Set the electrometer ATTENUATOR control to the infinity (∞) position. Adjust the recorder pen for mechanical and electrical zero.

3) Set the electrometer RANGE control to the BAL position.

4) Set the ATTENUATOR control to the 1 position.

5) Use the electrometer BALANCE control to set the recorder pen at electrical zero.

6) Set the RANGE control to the EC X $10^{-9}$ position (1 X $10^{-9}$ A/mV).

7) Set the ATTENUATOR control to the 2 position (2 X $10^{-9}$ A/mV).

8) Set the electrometer CELL VOLTAGE control to the EC position.

9) Use the electrometer BUCKING control to set the recorder pen at electrical zero.

10) Set the CELL VOLTAGE control to the OFF position. The observed recorder pen deflection is the $^{63}$Ni ECD standing current (% deflection X ATTENUATOR setting X RANGE setting). The standing current should be $\geq$ 1 X $10^{-9}$ A (50% X 2 X $10^{-9}$) which is a 50% deflection of the recorder pen at these settings. If the standing current is less, refer to Section 5 of this manual.

NOTE

**The recorder pen deflection is actually a measurement of the bucking voltage required to offset the detector standing current. The bucking voltage, rather than the standing current, is measured to eliminate the necessity of reversing the recorder leads.**

# SECTION 5
# SERVICE AND MAINTENANCE

## 5.1 GENERAL

This section of the manual comprises an operator troubleshooting chart and necessary detector maintenance procedures.

## 5.2 OPERATOR TROUBLESHOOTING

Some of the more common problems encountered with the $^{63}Ni$ ECD are listed in Table 5-1. If your detector appears faulty, locate the symptom in Table 5-1 and follow the suggested corrective measures. If the problem can not be corrected, contact your Varian Aerograph Sales or Service Representative.

TABLE 5-1
$^{63}Ni$ ECD OPERATOR TROUBLESHOOTING CHART

| Symptom | Possible Cause | Check and/or Remedy |
|---|---|---|
| No standing current. | Signal and cell voltage cables not connected. | Ensure detector electrodes and cables are installed as described in paragraph 2.5. |
| | Cell voltage shorted to ground. | Ensure detector exit tube (cell assembly cap) is not touching the tower cap. |
| | Septum too tight, obstructing carrier gas flow. | Tighten injector nut just enough to prevent leaks. |
| | Hydrogen supply not disconnected and capped off from instrument. | Hydrogen or air leaking into the chromatographic system will quench the standing current. Refer to Step (19) in paragraph 2.5.1 or 2.5.2. |
| Low standing current. | Leak in chromatographic system. | Leak check the chromatographic system as described in paragraph 4.3 step (20). |
| | Detector electrodes improperly installed. | Ensure electrodes are installed as described in paragraph 2.5. |
| | Contaminants in the carrier gas. | Only use high-purity nitrogen gas (oxygen and water vapor free). Refer to paragraph 3.3.5. |

TABLE 5-1 $^{63}$Ni ECD OPERATOR TROUBLESHOOTING CHART (Cont'd)

| Symptom | Possible Cause | Check and/or Rememdy |
|---|---|---|
| Low standing current (cont'd). | Contaminants in the sample. | Clean up the sample (i.e., extract lipids, pigments, etc). |
| | Contaminated chromatographic system. | Clean the chromatographic system as described in paragraph 4.3 and the detector as described in paragraph 5.3. |
| Normal standing current — low sensitivity. | Contaminants in the carrier gas. | Only use high-purity nitrogen gas (oxygen and water vapor free). Refer to paragraph 3.3.5. |
| | Contaminants in the sample. | Clean up the sample (i.e., extract lipids, pigments, etc). |
| | Contaminated chromatographic system. | Clean the chromatographic system as described in paragraph 4.3 and the detector as described in paragraph 5.3. |
| High standing current — low sensitivity. | Interelectrode-glass insulator conductance. | Foil temperature too high. Refer to paragraphs 3.3.2, 3.3.3, 3.3.4, and 3.3.6 |
| | Contaminants in the carrier gas. | Only use high-purity nitrogen gas (oxygen and water vapor free). Refer to paragraph 3.3.5. |
| | Contaminants in the sample. | Clean up the sample (i.e., extract lipids, pigments, etc). |
| | Contaminated chromatographic system. | Clean up the chromatographic system as described in paragraph 4.3 and the detector as described in paragraph 5.3. |
| Standing current sensitive to changes in the carrier gas flow rate. | Leak in chromatographic system. | Leak check chromatographic system as described in paragraph 4.3, Step (20). |
| | Contaminated chromatographic system. | Clean the chromatographic system as described in paragraph 4.3 and the detector as described in paragraph 5.3. |
| Unstable baseline. | Detector temperature has not equilibrated. | Allow the detector temperature to stabilize. |
| | Abrupt changes in the environmental temperature. | Ensure that the detector is not located near an air conditioning vent, etc. Cover exposed portion of detector with insulation. |
| | Detector temperature above 300°C. | Detector becomes unstable at high temperatures. Refer to paragraphs 3.3.2, 3.3.3, 3.3.4, 3.3.6, and 4.2.7. |
| | Contaminated chromatographic system. | Clean the chromatographic system as described in paragraph 4.3 and the detector as described in paragraph 5.3. |

TABLE 5-1 $^{63}$Ni ECD OPERATOR TROUBLESHOOTING CHART (Cont'd)

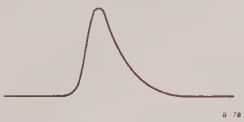
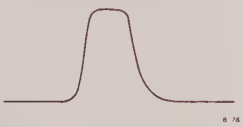
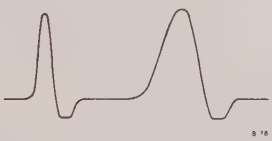
| Symptom | Possible Cause | Check and/or Rememdy |
|---|---|---|
| Tailing peaks.  | Column-oven temperature too low. | Increase column-oven temperature. |
| | Poor sample injection technique. | Refer to syringe manufacturer's instruction sheet. |
| | Sample interacting with liquid phase or solid support. | Ensure correct column has been selected for the type of analysis. |
| | Carrier gas flow rate too slow. | Increase carrier gas flow rate. |
| | Leak in chromatographic system. | Leak check chromatographic system as described in paragraph 4.3, step (20). |
| Rounded-off peaks.  | Operating out of the detector linear range. | Reduce sample size. |
| Negative dip after peaks.  | Contaminated detector. | Clean detector as described in paragraph 5.3. |
| Low sensitivity with normal retention time. | Insufficient sample size. A | Increase sample size. |
| | Attenuation too high. | Decrease electrometer ATTENUATOR setting. |
| | Leak in chromatographic system. | Leak check chromatographic system as described in paragraph 4.3, Step (20). |
| | Contaminated detector. | Clean detector as described in paragraph 5.3. |
| | Contaminated column. | Replace or recondition column. |
| | Contaminated carrier gas. | Replace molecular sieve. Ensure high-purity nitrogen is being used as described in paragraph 3.3.5. |
| | Decomposition of sample. | Ensure correct column, temperatures, and apparatus have been selected for the type of analysis. |
| Low sensitivity with long retention time. | Leak in chromatographic system. | Leak check chromatographic system as described in paragraph 4.3, Step (20). |
| | Carrier gas flow rate too low. | Increase carrier gas flow rate. |
| | Carrier gas flow system obstructed. | Check for foreign materials in gas flow system. Ensure septum is not obstructing carrier gas flow. |

TABLE 5-1 $^{63}$Ni ECD OPERATOR TROUBLESHOOTING CHART (Cont'd)

| Symptom | Possible Cause | Check and/or Rememdy |
|---|---|---|
| Sawtooth baseline. | Defective diaphragm in carrier gas flow controller. | Replace diaphram or flow controller. |
| Baseline drifting upscale. | Standing current decreasing because of column bleed.<br><br>Column, injector, or detector temperature changing. | Replace or recondition column.<br><br>Ensure column, injector and detector temperatures have stabilized. |
| Baseline drifting downscale. | Standing current increasing. | Good. Standing current may increase for several hours after installing the detector. |
| Sinusoidal Baseline. | COLUMN SAFETY LIMIT control being used as temperature controller.<br><br>Detector-base temperature cycling.<br><br>Column-oven door not properly closed.<br><br>Faulty column-oven temperature controller, especially with high-bleed columns. | Use as high-limit cutoff only. Use column-oven controller to set the column-oven temperature.<br><br>Faulty detector temperature controller. Refer to paragraph 4.2.7.<br><br>Ensure column-oven door seals when closed.<br><br>Replace column-oven temperature controller. |

## 5.3 CLEANING THE $^{63}$Ni ECD

There are two methods of cleaning the $^{63}$Ni ECD: Superheated steam cleaning and high-temperature boil off. The superheated steam cleaning procedure is the quickest; however, if ineffective, follow up with the high-temperature boiloff procedure.

### 5.3.1 Superheated Steam Cleaning

1) Turn off power to the instrument. Allow the column oven, detector, and injector to cool until their components can be handled.

2) Turn off the carrier gas and remove the analytical column.

3) Install an empty column and reestablish the carrier gas flow rate.

— CAUTION —

**Do not use columns with Teflon ferrules when injector, column, or detector temperatures exceed 250°C. The Teflon ferrules begin to flow at temperatures above 250°C.**

4) Set the column oven, detector, and injector temperatures at 300°C.

5) Make 5 injections of 100 µl of distilled water.

6) Cool the column oven, detector, and injector. Remove the empty column and reinstall the analytical column.

NOTE

**More specific data on superheated steam cleaning is described in an article by I. H. Williams, "Note on the Cleaning of the $^{63}$Ni Electron Capture Detector", in the Journal of the Association of Official Analytical Chemists, 51 (1968) 64.**

### 5.3.2 High-Temperature Boiloff Cleaning

1) Turn off power to the instrument. Allow the column oven, detector, and injector to cool until their components can be handled.

2) Turn off the carrier gas and remove the analytical column.

3) Install an empty column and reestablish the carrier gas flow rate.

— CAUTION —

**Do not use columns with Teflon ferrules when injector, column, or detector temperatures exceed 250°C. The Teflon ferrules begin to flow at temperatures above 250°C.**

4) Set the column oven, detector, and injector temperatures at 400°C.

5) Allow a minimum of 8 hours for the chromatographic system to bake out.

6) Cool the column oven, detector, and injector. Remove the empty column and reinstall the analytical column.

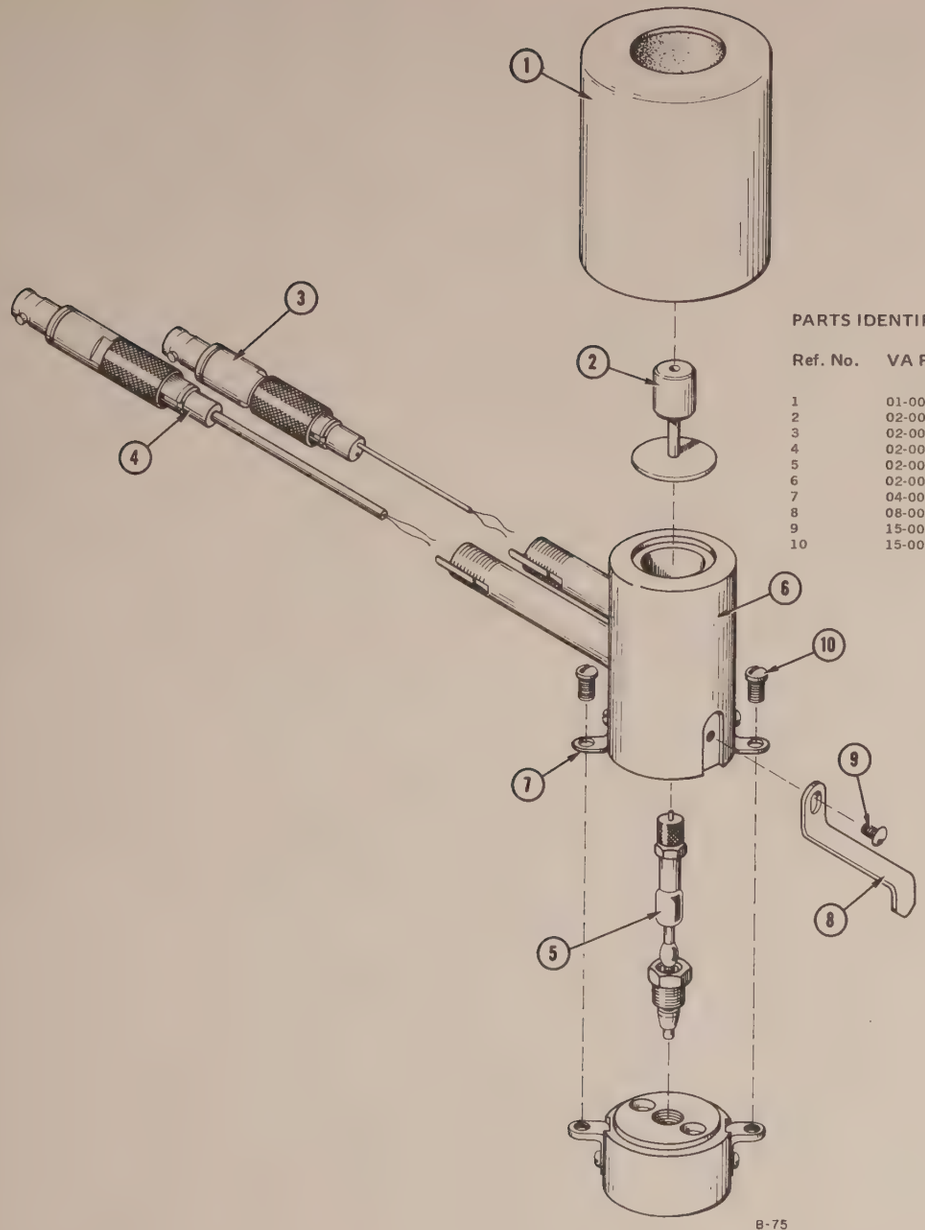
# SECTION 6
# ILLUSTRATED PARTS LISTS

## 6.1 GENERAL

This section of the manual comprises the $^{63}Ni$ ECD Kit Parts Identification Drawings and Parts List. The parts are identified on the drawing by a reference number within a circle. The reference number is listed numerically in the parts list. The number is followed by a corresponding part number and material description. Use the ten-digit part number and material description when ordering replacement parts. Also, when ordering parts, include the complete Model and Serial number of your instrument.

Listed below are the titles, drawing numbers and page numbers of the Parts Identification Drawings in this manual.

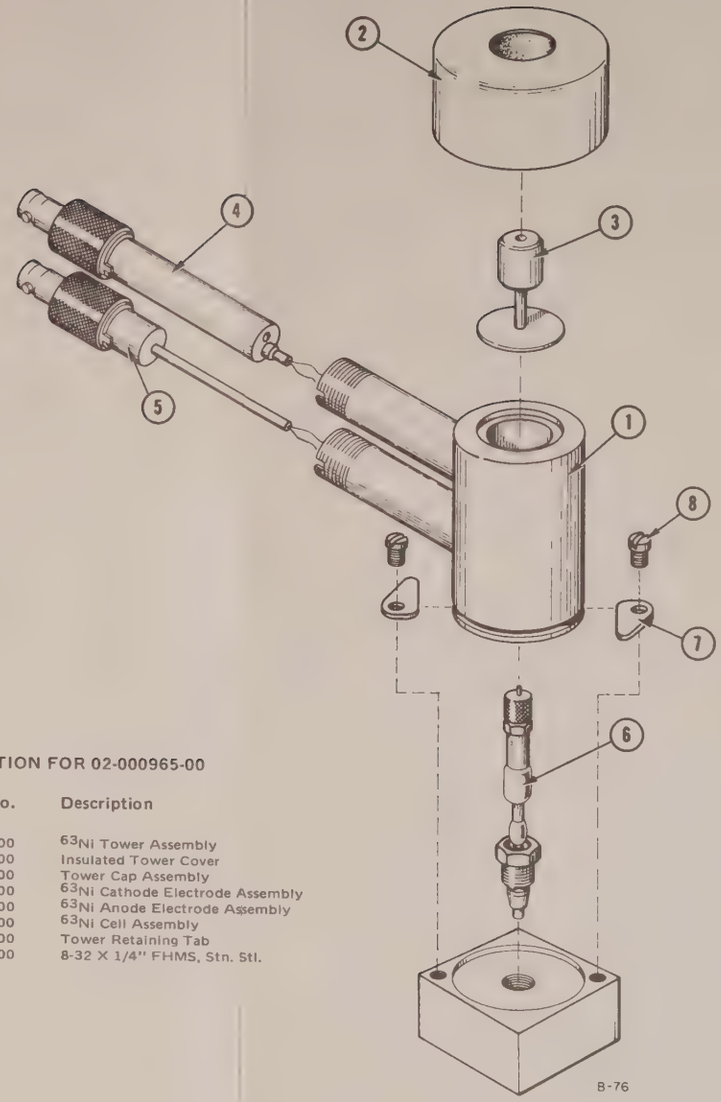

| TITLE | DRAWING NO. | PAGE NO. |
|---|---|---|
| $^{63}Ni$ ECD Kit for Series 1200 and 2100 Instruments | 02-000770-00 | 6-2 |
| $^{63}Ni$ ECD Kit for Series 1400, 1700, 1800, 2700, and 2800 Instruments | 02-000965-00 | 6-2 |




PARTS IDENTIFICATION FOR 02-000770-00

| Ref. No. | VA Part No. | Description |
|---|---|---|
| 1 | 01-000699-00 | Insulated Tower Cover |
| 2 | 02-000594-00 | Tower Cap Assembly |
| 3 | 02-000647-00 | $^{63}$Ni Cathode Electrode Assembly |
| 4 | 02-000648-00 | $^{63}$Ni Anode Electrode Assembly |
| 5 | 02-000969-00 | $^{63}$Ni Cell Assembly |
| 6 | 02-001075-00 | $^{63}$Ni Tower Assembly |
| 7 | 04-000221-00 | Tower Mounting Bracket |
| 8 | 08-000068-00 | Temperature-Limit-Switch Actuator Arm |
| 9 | 15-000002-00 | 6-32 X 1/4" RHMS |
| 10 | 15-000153-00 | 8-32 X 1/4" PHMS, Stn. Stl. |

$^{63}$Ni ECD KIT FOR 1200 AND 2100 SERIES INSTRUMENTS (02-000770-00)




PARTS IDENTIFICATION FOR 02-000965-00

| Ref. No. | VA Part No. | Description |
|---|---|---|
| 1 | 01-000683-00 | $^{63}$Ni Tower Assembly |
| 2 | 01-000693-00 | Insulated Tower Cover |
| 3 | 02-000594-00 | Tower Cap Assembly |
| 4 | 02-000954-00 | $^{63}$Ni Cathode Electrode Assembly |
| 5 | 02-000958-00 | $^{63}$Ni Anode Electrode Assembly |
| 6 | 02-000969-00 | $^{63}$Ni Cell Assembly |
| 7 | 08-000072-00 | Tower Retaining Tab |
| 8 | 15-004722-00 | 8-32 X 1/4" FHMS, Stn. Stl. |

$^{63}$Ni ECD KIT FOR 1400, 1700, 1800, 2700, AND 2800 SERIES INSTRUMENTS (02-000965-00)

varian aerograph

2700 mitchell drive/walnut creek/california 94598/415-939-2400
specialists in chromatography and data handling systems

# ROTAMETER GAS FLOW CONTROLLER

# INSTRUCTION MANUAL

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Manufactured in the United States of America

PUBLICATION NO. 85-001118-08 PRINTED IN U.S.A. OCTOBER 1970

## SAFETY PRECAUTIONS AND OPERATING INSTRUCTIONS

This instruction manual is provided to help you establish operating conditions which will permit efficient use of your equipment. You will notice that the manual describes special considerations and precautions in the form of notes, cautions, and warnings, that not only aid you in obtaining the best performance from your equipment but in calling to your attention potential hazards (e.g., electrical, mechanical, etc.) that might cause personal injury, and how to avoid these hazards. It is important, therefore, that you operate your equipment in accordance with this instruction manual and additional information which may be provided by Varian Instrument Division, Aerograph® Products. Address any questions regarding the safe and proper use of your instrument to:

Varian Instrument Division, Aerograph® Products
611 Hansen Way
Palo Alto, California 94303

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## SPARE PARTS AVAILABILITY

It is the policy of the Varian Instrument Division to provide operational spare parts for any instrument and major accessory for a period of five (5) years after shipment of the final production run of that instrument. Spare parts will be available after this five (5) year period but on an "as available" basis.

Operational spare parts are defined as those individual electrical or mechanical parts that are susceptible to failure during their normal operation. Examples include resistors, inductors, capacitors, semi-conductor devices of all kinds, relays, lamps, temperature probes, detector elements, motors, etc.

Sheet metal parts, structural members or assemblies, and castings, printed circuit cards, and functional modules are normally capable of being rebuilt to like-new condition throughout their useful life and therefore will be supplied on an "as available" basis only after the final production run of the instrument.

The repair and/or reconditioning of instruments and accessories is handled on a time and materials basis after expiration of the Warranty period. Please contact your nearest Varian Instrument Division Sales and Service Office for a quotation.

6/20/1972

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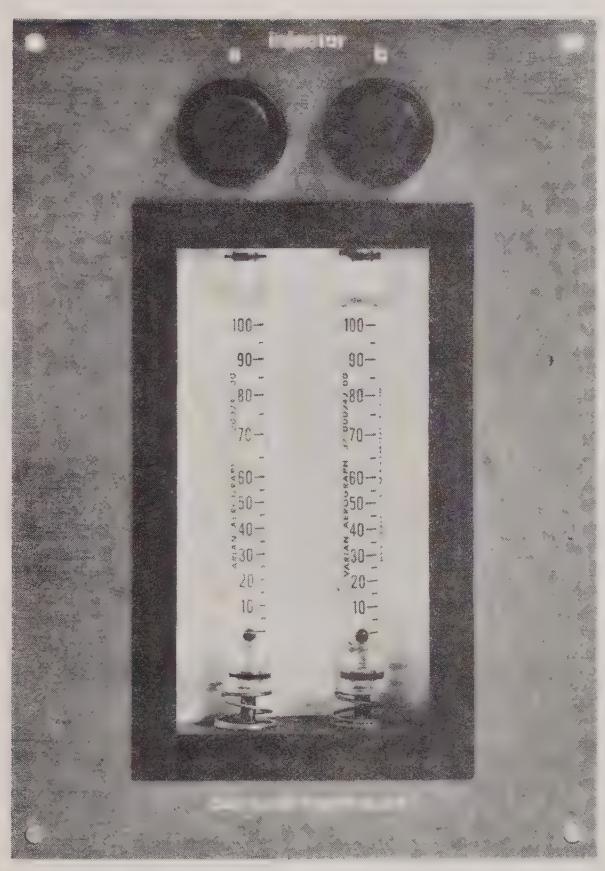
# SECTION 1
# INTRODUCTION



FLOWMETER MODULE

## 1.1 GENERAL

The Varian Aerograph™ Rotameter Gas Flow Controller Module is designed for use in the 2700 and 2800 Series Gas Chromatographs. Comprising two differential flow controllers, two precision needle valves and two rotameters (one for each flow system), the flow controllers maintain a constant carrier gas flow rate in the column flow system of the instrument and the rotameters monitor relative flow rate of the flow system. Refer to Table 1-1 for Rotameter Gas Flow Controller Module specifications.

**TABLE 1-1**
**ROTAMETER GAS FLOW CONTROLLER MODULE SPECIFICATIONS**

**Working Pressure:**
200 psi (max) regulated.

**Flow Range:**
10 to 55 ml/min with standard rotameters.
50 to 150 ml/min with optional rotameters.

**Flow Accuracy:**
2% of set point.

**Difference Between Inlet/Outlet Pressure for Constant Output:**
$\leqslant$ 1/2 input pressure.

# SECTION 2
# OPERATION

## 2.1 GENERAL

The Rotameter Gas Flow Controller Module is optional for all 2700 and 2800 Series instruments. When ordered with the instrument, the module will be installed in the instrument before shipment.

The standard rotameters supplied with the module have a range of approximately 10 to 55 ml/min. However, a set of 50 to 150 ml/min (VA Part No. 37-000242-00) rotameters are available and are directly interchangeable with the 10 to 55 ml/min rotameters. The 50 to 150 ml/min rotameters are supplied with the module when specified and should be used where operation requiring high column flow rates are anticipated.

To familiarize yourself with the location and functional description of the controls on the module, refer to Figure 2-1 and accompanying description of controls.

## 2.2 CALIBRATION OF ROTAMETER GAS FLOW CONTROLLER MODULE

The flowmeter module can handle 10 to 55 ml/min rotameters or 50 to 150 ml/min rotameters. Longer columns or columns packed with very fine mesh supports create a greater back pressure through the column. This increased back pressure causes the float in the rotameter to reside at a relatively lower level. The use of very short columns or columns packed with a coarse mesh support will normally cause the float to reside at or near the top of the scale.

Each rotameter is labeled from 0 to 100 in increments of 5. These are reference marks only and should be used in calibrating the rotameters. The rotameters must be calibrated individually each time a different type of carrier gas is used. Calibration records should be made for each rotameter and used when adjusting flow rates.

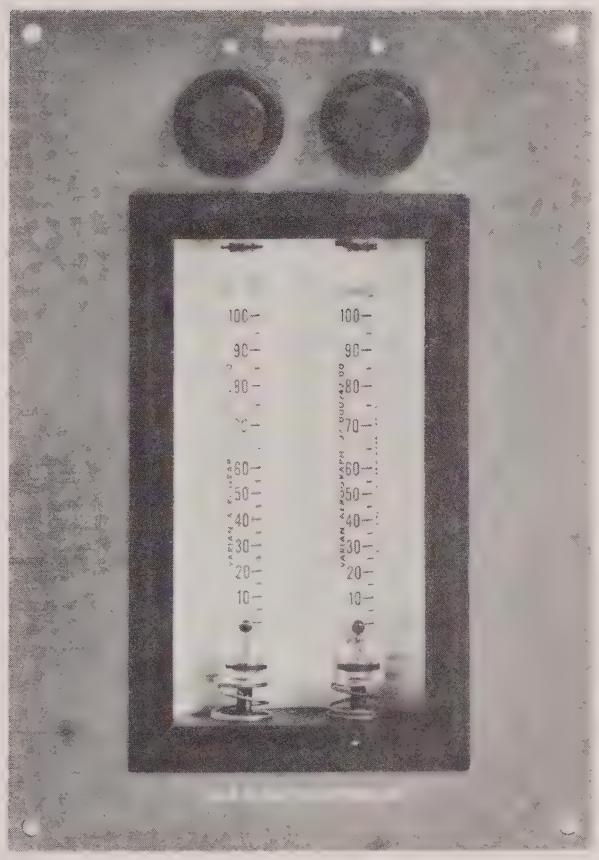



FIGURE 2-1 FLOWMETER MODULE

Rotameters
: Indicates relative flow rate of carrier gas through respective injector-column-detector flow system. Meters must be calibrated before being used for each set of columns and operating conditions.

INJECTOR A and B Controls
: These two controls set flow rate as indicated by rotameter for respective flow system. INJECTOR A controls column A flow system; INJECTOR B controls column B flow system.

Any grease, oil or foreign material in the rotameter tubes will cause the float to stick or read erroneously.

Both flow systems (A and B) are calibrated in the same manner. Before proceeding with the calibration procedures, make sure column(s) is installed and associated flow system leak checked in the basic instrument. Calibrate module as follows:

1) Adjust regulator on carrier gas cylinder for at least 65 psi. Allow pressure to stabilize.

NOTE

**The flow controllers will not operate properly if the tank pressure is less than two times the pressure drop of the column. Sixty-five psi tank pressure is adequate for most operating conditions. If extra long columns are used with exceptional high pressure drops, even higher tank pressures may be required.**

2) Turn CARRIER GAS FLOW control (located on main control panel of instrument) fully counterclockwise (on).

3) Adjust INJECTOR A control valve to obtain a reading of "10" on rotameter. Tap meter gently to make sure bead returns to proper level. Readjust control valve as required. Measure flow rate of carrier gas with soapbubble flowmeter and record reading for flowmeter setting of "10". Soapbubble flowmeter is supplied with basic instrument. Connect soapbubble flowmeter to source being monitored, e.g., end of column, at ionization detector, etc.

— CAUTION —

**Injector A and/or B are control valves, not shut-off valves. Never tighten to full clockwise position.**

4) Repeat step (3) for each increase of 10 on rotameter until maximum flow rate is reached.

5) Calibrate INJECTOR B as outlined in steps (3) and (4).

## 2.3 OPERATION

Operation of the module is quite straightforward. Refer to the conditions established in calibrating the module (paragraph 2.2) and adjust INJECTOR A and/or B controls to obtain the desired indication on the appropriate rotameter and thus the desired carrier gas flow rate.

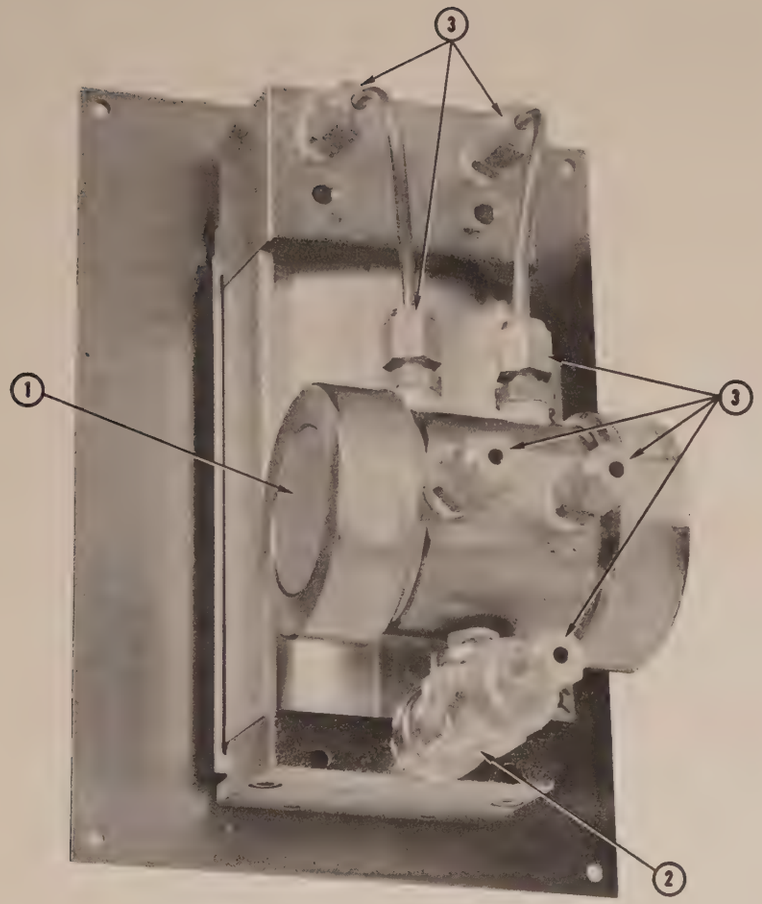
# SECTION 3
# ILLUSTRATED PARTS LIST

## 3.1 GENERAL

This section of the manual comprises an illustrated parts list for the Rotameter Gas Flow Controller Module.

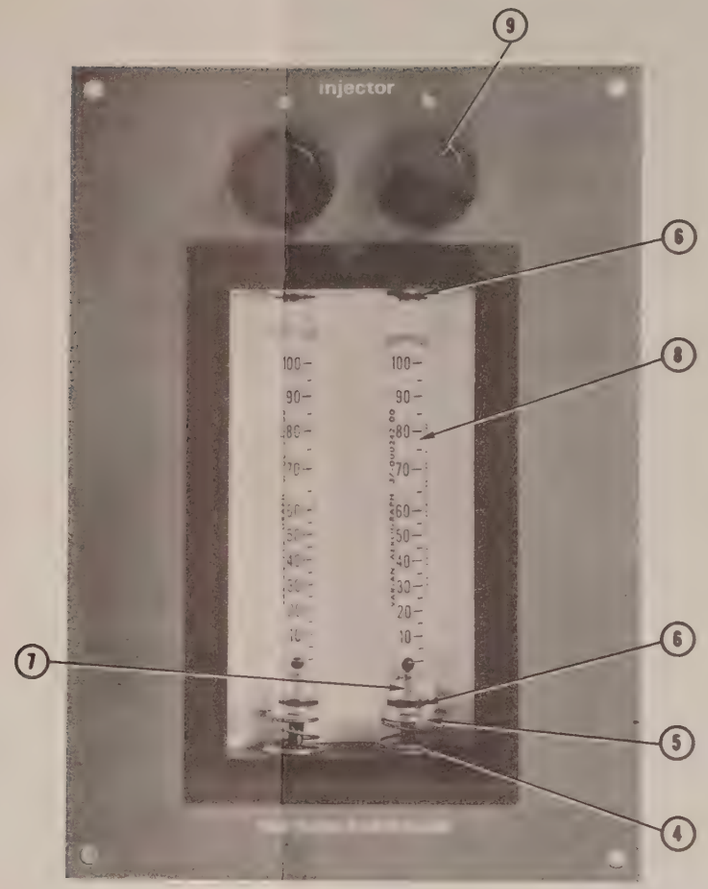
Listed below is the title and drawing number of the illustrated parts list.


| DWG. TITLE | DWG. NO. | PAGE |
|---|---|---|
| Rotameter Flow Controller Module | 02-000757-00 | 3-2 |




MODULE REAR VIEW

| Item | Part No. | Description |
|---|---|---|
| 1 | 57-000038-00 | Diff. Flow Controller |
| 2 | 16-000334-00 | Branch Tee, 1/8 in BR |
| 3 | 16-000040-00 | Connector, 1/8 to 1/8 M.P.T. |
| 4 | 46-000018-00 | Meter Tube Retaining Spring |
| 5 | 15-000753-00 | Wash, Ret Flowmeter Tube |
| 6 | 69-000008-00 | O-Ring, Neo 1/8 ID X 1/4 OD |
| 7 | 46-000019-00 | Meter Float Retaining Spring |
| 8 | 37-000203-00 | Rotameter Tubes |
| 9 | 57-000042-00 | Fine Metering Cartridge Valve |




MODULE FRONT VIEW

ROTAMETER FLOW CONTROLLER MODULE

varian aerograph

2700 mitchell drive/walnut creek/california 94598/415-939-2400
specialists in chromatography and data handling systems

# PRESSURE GAUGE GAS FLOW CONTROLLER

# INSTRUCTION MANUAL

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Manufactured in the United States of America

PUBLICATION NO. 85-001118-09 PRINTED IN U.S.A. OCTOBER 1970

## SAFETY PRECAUTIONS AND OPERATING INSTRUCTIONS

This instruction manual is provided to help you establish operating conditions which will permit efficient use of your equipment. You will notice that the manual describes special considerations and precautions in the form of notes, cautions, and warnings, that not only aid you in obtaining the best performance from your equipment but in calling to your attention potential hazards (e.g., electrical, mechanical, etc.) that might cause personal injury, and how to avoid these hazards. It is important, therefore, that you operate your equipment in accordance with this instruction manual and additional information which may be provided by Varian Instrument Division, Aerograph® Products. Address any questions regarding the safe and proper use of your instrument to:

Varian Instrument Division, Aerograph® Products
611 Hansen Way
Palo Alto, California 94303

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## SPARE PARTS AVAILABILITY

It is the policy of the Varian Instrument Division to provide operational spare parts for any instrument and major accessory for a period of five (5) years after shipment of the final production run of that instrument. Spare parts will be available after this five (5) year period but on an "as available" basis.

Operational spare parts are defined as those individual electrical or mechanical parts that are susceptible to failure during their normal operation. Examples include resistors, inductors, capacitors, semi-conductor devices of all kinds, relays, lamps, temperature probes, detector elements, motors, etc.

Sheet metal parts, structural members or assemblies, and castings, printed circuit cards, and functional modules are normally capable of being rebuilt to like-new condition throughout their useful life and therefore will be supplied on an "as available" basis only after the final production run of the instrument.

The repair and/or reconditioning of instruments and accessories is handled on a time and materials basis after expiration of the Warranty period. Please contact your nearest Varian Instrument Division Sales and Service Office for a quotation.

6/20/1972

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# SECTION 1
# INTRODUCTION

## 1.1 GENERAL

The Varian Aerograph™ Pressure Gauge Gas Flow Controller Module is designed for use in the 2700 and 2800 Series Gas Chromatographs. Comprising two differential flow controllers, two pressure gauges and two precision needle valves (one for each flow system), the flow controllers maintain a constant carrier gas flow rate in the column flow system of the instrument and the pressure gauges monitor the back pressure of the flow system. Refer to Table 1-1 for Pressure Gauge Module specifications.

TABLE 1-1
PRESSURE GAUGE MODULE SPECIFICATIONS

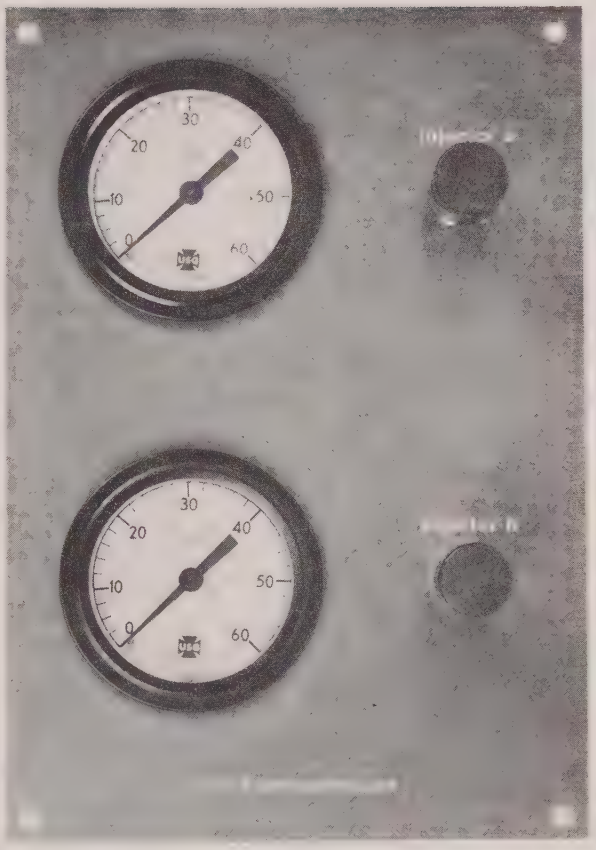
**Working Pressure:**
200 psi (max) regulated

**Flow Range:**
2 ml/min to 1000 ml/min

**Flow Accuracy:**
1% of set point

**Difference Between Inlet/Outlet Pressure For Constant Output:**
15 psi (min) input pressure

**Pressure Gauge Range:**
0 to 60 psi




PRESSURE GAUGE MODULE

# SECTION 2
# OPERATION

## 2.1 GENERAL

The Pressure Gauge Module is standard on all 2700 and 2800 Series instruments. The module is installed in the instrument before shipment.

To familiarize yourself with the location and functional description of the controls on the module, refer to Figure 2-1 and accompanying description of controls.

## 2.2 CALIBRATION OF PRESSURE GAUGE GAS FLOW CONTROLLER MODULE

The pressure gauge module requires calibration before being used. Once calibrated for a specific column, head pressure, etc., you can refer to gauge to determine carrier gas flow rate.

Both flow systems (A and B) are calibrated in the same manner. Before proceeding with the calibration procedures, make sure column(s) is installed and associated flow system leak checked. Calibrate pressure gauge module as follows:

1) Adjust regulator on carrier gas cylinder for at least 65 psi. Allow pressure to stabilize.

NOTE

**The flow controllers will not operate properly if the tank pressure is less than 15 lbs. over the pressure drop of the column. Sixty-five psi tank pressure is adequate for most operating conditions. If extra long columns are used with exceptional high pressure drops, even higher tank pressures may be required.**

2) Turn CARRIER GAS FLOW control (located on main control panel) fully counterclockwise (on).

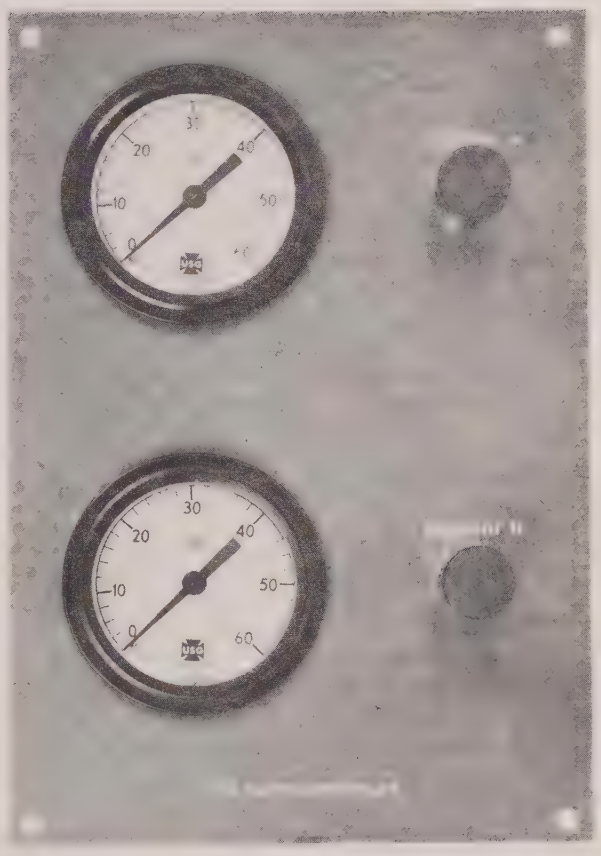



FIGURE 2-1 PRESSURE GAUGE MODULE

PRESSURE Gauge
Monitors column back pressure of respective flow system.
Gauge readings should be noted for each set of columns and operating conditions for future flow rate references.

INJECTOR A and B
These two controls set flow rates for respective flow system. INJECTOR A controls column A flow system; INJECTOR B controls column B flow system.

3) Adjust INJECTOR A control valve to obtain desired flow rate. Measure flow rate of carrier gas with soapbubble flowmeter and record reading indicated on pressure gauge. Soapbubble flowmeter is supplied with basic instrument. Connect soapbubble flowmeter to source being monitored, e.g., end of column, at ionization detector, etc.

— CAUTION —

**Injector A and B are control valves, not shut-off valves. Never tighten to full clockwise position.**

4) Readjust INJECTOR A control to obtain half the desired flow rate and twice the desired flow rate. Monitor flow rate (3) and the pressure readings obtained in steps (3). From this data, a graph may be prepared which can be used as a guide in the future for adjustment of flow rate; however, the same operating conditions, i.e., tank head pressure, column, must be used. Each time you change to a different column, change tank pressure, etc., you must recalibrate the pressure gauges.

5) Repeat steps (3) and (4) to calibrate INJECTOR B flow system.

## 2.3 OPERATION

Operation of the module is quite straightforward. Refer to the conditions established in calibrating the module (paragraph 2.2) and adjust INJECTOR A and/or B controls to obtain the desired pressure as indicated on the pressure gauge and thus the desired carrier gas flow rate.

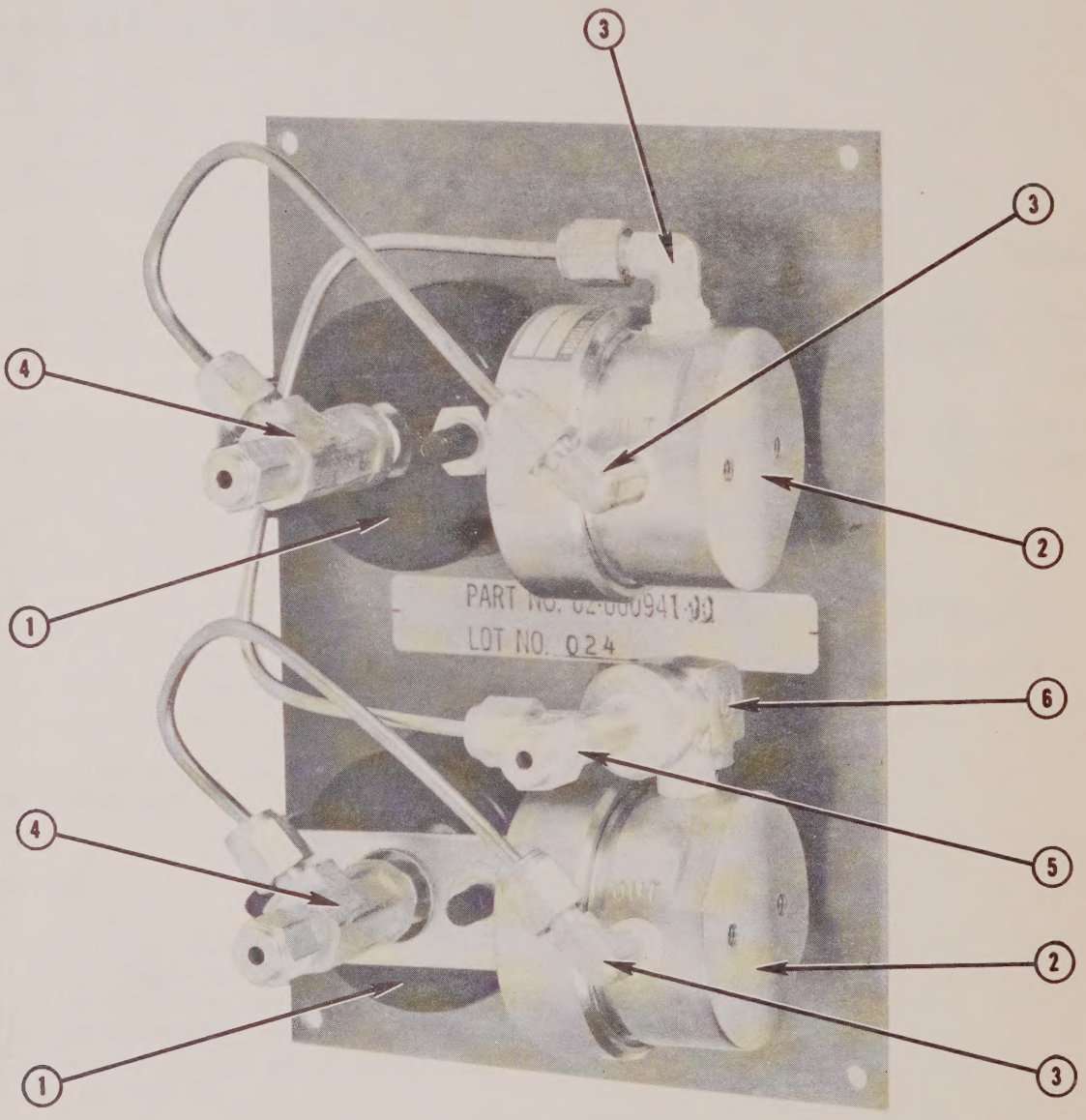
# SECTION 3
# ILLUSTRATED PARTS LIST

## 3.1 GENERAL

This section of the manual comprises an illustrated parts list for the Pressure Gauge Gas Flow Control Module.

Listed below is the title and drawing number of the illustrated parts list.


| DWG. TITLE | DWG. NO. | PAGE |
|---|---|---|
| Pressure Gauge Flow Controller | 02-000941-00 | 3-2 |




PRESSURE GAUGE FLOW CONTROLLER

| Item No. | Parts List No. | Description |
|---|---|---|
| 1 | 29-000041-00 | Gauge — Pressure, 0-60 PSIG |
| 2 | 57-000122-00 | Valve-Back Reference Flow Controller |
| 3 | 16-000137-00 | Elbow — Male, 1/8" |
| 4 | 16-000383-00 | Tee — Female Run, 1/8" |
| 5 | 16-000138-00 | Tee — Male Run, 1/8" |
| 6 | 16-000384-00 | Elbow — Street, 1/8" |

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